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Bureau of Mines Research on Resource Recovery

Reclamation, Utilization, Disposal, and Stabilization

UNITED STATES DEPARTMENT OF THE INTERIOR

Information Circular 8750

Bureau of Mines Research on Resource Recovery

Reclamation, Utilization, Disposal, and Stabilization

By Max J. Spendlove

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BUREAU OF MINES RESEARCH ON RESOURCE RECOVERY

Reclamation, Utilization, Disposal, and Stabilization

by

Max J. Spendlove[1]

ABSTRACT

The Federal Bureau of Mines has been a leader in resource recovery research since it was founded in 1910. Until the early 1960's, most of the projects were concerned primarily with the task of extending and improving conventional technology oriented to secondary metals and minerals upgrading and processing for marketable products of highest value. By the mid-1960's with the passage of the Solid Waste Disposal Act of 1965, all resource recovery research programs were increased in scope and intensity as an effort to solve the growing number of environmental problems and the rapid depletion of domestic energy and mineral resources. Many new materials were investigated including urban refuse, manufacturing, industrial wastes, and stockyard wastes. The objective was to promote maximum recycling as the most practical answer to problems of waste disposal, pollution control, and the short supply of minerals and energy. The new research projects ranged from the conversion of animal manure and other organics to oil, to the recovery of precious and common metals from demolished aircraft and electronic scrap. This profile includes brief descriptions of many selected research projects that typify the total spectrum of Bureau of Mines research in the important field of resource recovery.

INTRODUCTION

Background

Most of our modern societies' wastes are generated during the production of the millions of materials and devices that characterize the prevailing life styles. Such wastes occur during the production of all consumer materials ranging from ballpoint pens and mousetraps to atomic reactors and spaceships. It is, in fact, difficult to think of any consumer item that can be made without the concurrent generation of wastes at nearly every phase of its production. Not only do these production wastes contribute to a massive total accumulation, but most of the consumer items themselves eventually become part of that total after they have been used and discarded. Collectively, all wastes and discarded consumer goods comprise a vast rejected resource, part of which still contains essentially all of the material values that went into the original production. Although most of this rejected resource has only a minor potential for exploitation, the discarded goods and materials fraction comprises a significant exploitable manmade reserve of metals, minerals, and organic materials. The Bureau of Mines, through its long-range innovative resource-recovery research programs, has, in fact, demonstrated the considerable potential for exploiting these materials.

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[1] Consulting metallurgist, College Park Metallurgy Research Center, College Park, Md.

Historically, the practice of resource recovery has followed the quasi-natural trend in which society does not become seriously alarmed about its resources until they are in short supply, nor does society become alarmed about the general environment until it becomes uncomfortable or threatening. The level of related research activity has essentially paralleled societies' demands for natural resource raw materials, a trend that has been evident during the advance of civilization through all of the material epochs--stone, bronze, and iron ages--and on through the epochs of achievement--the industrial, nuclear, and space ages. However, only during the last decade has the widespread public concern for the environment exceeded its concern for exploiting domestic resources. Now, at its highest level of intellectual, technological, and social development, society has the best opportunity, the greatest capability, and the most pressing need to recover the maximum possible values from its wastes and spent resources, and concurrently improve environmental quality significantly.

Except in times of war, the problems attending resource recovery were formerly considered relatively minor until the general public became better informed about the rapidly diminishing domestic mineral reserves, the steady deterioration of the environment, and the rising costs of waste disposal by nonpolluting methods.

In contrast with former public indifference toward resource recovery and the environment, the Bureau of Mines has always maintained an interest in these technologies and included related research projects as essential elements of its long-range programs for maintaining national supplies of minerals and metals. The Bureau has, in fact, included resource recovery and antipollution projects in its research programs for 66 years. Until the early 1960's, a moderate research effort was directed primarily toward extending conventional technology to recover secondary materials; however, during the mid-1960's, the research programs were increased in scope and intensity as part of a new concerted effort to solve some of the most acute problems of minerals supply, waste disposal, and pertinent antipollution control. Many different materials were included in these new investigations, such as stockyard wastes as a potential source of energy materials; urban refuse as a source of fuel, minerals, and metals; and a multitude of industrial and commercial wastes for whatever values might be recovered. The environmental aspects of these investigations were largely incidental to the more direct efforts aimed at increasing mineral supplies and reducing process wastes and waste disposal costs. Through its sustained activities in this field, the

Bureau's contributions to the technology have increased steadily since it was founded in 1910 (39).[2]

For the purpose of this report, the definition of resource recovery includes the collection of processing wastes and postconsumer materials and goods, and the development of separation, extraction, refining, and utilization processes essential for making marketable products. It also includes related waste disposal and environmental control operations essential for meeting current and anticipated statutory environmental and safety regulations. The quantities, compositions, and location of these rejected resource materials have, in recent years, been the subjects of numerous studies, publications, and speeches, most of which stress the potentials for exploitation or better disposal practice (3-4, 17, 77-79). In general, these potentials are directly related to the types of raw materials involved, the primary production methods originally employed, and the product end-use patterns, all of which tend to effect the successive transfer of values from incoming raw material supplies to outgoing products in each of the consecutive extraction, processing, manufacturing, and consuming sectors. Natural raw materials such as coal, petroleum, natural gas, and some chemical minerals are totally consumed by society and thus have no potential for resource recovery. The same applies for metals consumed in dissipative uses such as the zinc for galvanizing and the lead for tetraethyl lead in gasoline.

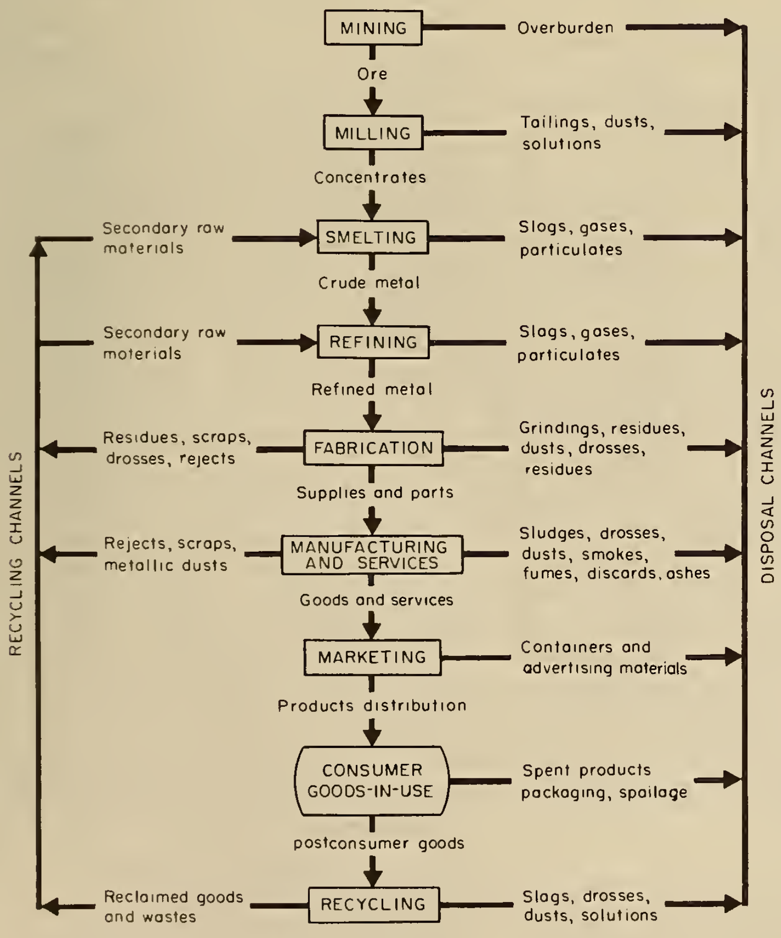
MINING
Overburden
Ore
MILLING
Tailings, dusts, solutions
Concentrates
Secondary raw materials
SMELTING
Slags, gases, particulates
Crude metal
Secondary raw materials
REFINING
Slags, gases, particulates
Refined metal
Residues, scraps, drosses, rejects
FABRICATION
Grindings, residues, dusts, drosses, residues
Supplies and parts
Rejects, scraps, metallic dusts
MANUFACTURING AND SERVICES
Sludges, drosses, dusts, smokes, fumes, discards, ashes
Goods and services
MARKETING
Containers and advertising materials
Products distribution
CONSUMER GOODS-IN-USE
Spent products packaging, spoilage
postconsumer goods
Reclaimed goods and wastes
RECYCLING
Slags, drosses, dusts, solutions
RECYCLING CHANNELS
DISPOSAL CHANNELS

FIGURE 1. - System of production, utilization, disposal, and recycling metal-containing products.

Origin of the Waste Materials Resource

The system illustrated in figure 1 is an example of the metal industry and consumer contributions to the national productivity and to the accumulation of mineral wastes. Other wastes are generated in similar patterns during the production of nonmetallic commodities such as plastics, fertilizers, lumber, textiles, foodstuffs, and thousands of other items.

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[2] Underlined numbers in parentheses refer to the list of references preceding the appendix.

Bureau of Mines research on these rejected waste materials was traditionally directed toward extending the technology of mineral extraction, utilization, and waste disposal, all of which comprise major elements in its minerals conservation program.

The purpose of this report is to present a summary profile of the many Bureau of Mines investigations that typify its traditional research activities on resource recovery, waste utilization, waste disposal, and mined land reclamation. Because of the very large number of Bureau series publications and outside papers by Bureau authors (39), only a representative number of research investigations are discussed here to reflect the nature, scope, and progress to date of the entire resource-recovery research program.

Discussions are arranged generally in groups according to the sequence of raw materials and product flow through the major sectors of the supply, consumption, and waste disposal system (fig. 2). Of particular importance in this illustration is the fact that most of the wastes generated in the first three sectors (extraction beneficiation, and processing) are moved directly to disposal sites. These are the raw materials processing residuals left after the supply materials have been stripped of their value constituents to make products. The magnitude of these combined wastes, in weight and volume, is tremendous, but the total of all recoverable values is relatively insignificant. Except in a very few and unique cases, these wastes present growing disposal problems rather than significant opportunities for resource recovery. Conversely, the total of all wastes generated in the last three sectors (manufacturing secondary-materials processing, and postconsumer utilization and disposal) is considerably smaller in weight and volume than the preceding processing wastes, but the potential for

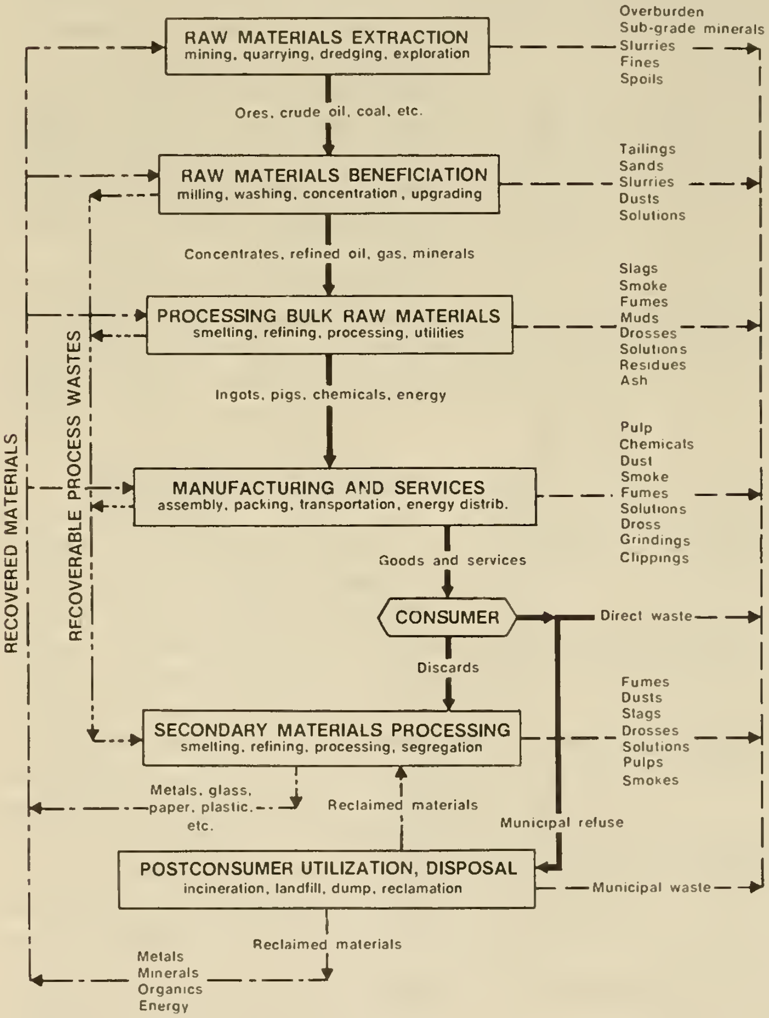
RAW MATERIALS EXTRACTION
mining, quarrying, dredging, exploration
Overburden
Sub-grade minerals
Slurries
Fines
Spoils
Ores, crude oil, coal, etc.
RAW MATERIALS BENEFICIATION
milling, washing, concentration, upgrading
Tailings
Sands
Slurries
Dusts
Solutions
Concentrates, refined oil, gas, minerals
PROCESSING BULK RAW MATERIALS
smelting, refining, processing, utilities
Slags
Smoke
Fumes
Muds
Drosses
Solutions
Residues
Ash
Ingots, pigs, chemicals, energy
MANUFACTURING AND SERVICES
assembly, packing, transportation, energy distrib.
Pulp
Chemicals
Dust
Smoke
Fumes
Solutions
Dross
Grindings
Clippings
Goods and services
CONSUMER
Direct waste
Discards
SECONDARY MATERIALS PROCESSING
smelting, refining, processing, segregation
Fumes
Dusts
Slags
Drosses
Solutions
Pulps
Smokes
Metals, glass, paper, plastic, etc.
Reclaimed materials
Municipal refuse
POSTCONSUMER UTILIZATION, DISPOSAL
incineration, landfill, dump, reclamation
Municipal waste
Reclaimed materials
Metals
Minerals
Organics
Energy
RECOVERED MATERIALS
RECOVERABLE PROCESS WASTES
UNRECOVERED WASTES DISPOSAL SITES

FIGURE 2. - Materials supply, utilization, and disposal sections.

recovering values is many times greater. The wastes from the manufacturing, secondary materials, and postconsumer sectors still contain nearly all of the material values that went into their original production in the preceding sectors. Collectively, the wastes generated in the last three sectors are known in the Bureau of Mines as urban ore, the most valuable part of the entire waste resource. The raw materials that supply the secondary materials industry comprise a particularly valuable resource, but these materials have been exploited extensively for centuries. The products of the secondary materials industry have, in fact, contributed significantly to supplying the annual national demand for paper, glass, metals, fabrics, and other materials for many decades. However, although the secondary materials industry is reasonably efficient, there are many opportunities for process improvement and better waste disposal practice. The same is true for the entire primary materials industry. Although the Bureau of Mines has conducted extensive research investigations in both primary and secondary mineral materials technology, the following discussions on resource recovery research are directed to selected typical investigations on specific industrial and consumer wastes and combination of wastes generated in the supply-consumption sectors shown in figure 2.

RAW MATERIALS EXTRACTION

The U.S. demand for new mineral supplies has continued to increase steadily for more than a century, having been stimulated by the steady population growth and the continuous rise in the standard of living. According to a recent study (13) by the Department of Interior's Bureau of Outdoor Recreation, in cooperation with the Bureau of Mines, one consequence of this demand for materials of mineral origin is an estimated 4 million acres of land disturbed by surface mining alone. The Bureau of Mines estimated that the acreage disturbed in 1964 exceeded 1 million acres. It was further estimated that mining-engendered operations added 160,000 acres of disturbed land occupied by wastes from underground mines, mineral processing plants, and the more than 290 burning refuse banks which, in themselves, occupied 3,200 acres in 1968. It is generally agreed that the total accumulation of solid wastes generated by the minerals extraction sector is so enormous that the data tend to lose practical meaning; however, the extent of these wastes can be illustrated by a map (fig. 3) that shows the location of 693 randomly selected mining sites visited during a U.S. Department of Interior study (98) on surface mining and the environment. There are at least 130 mining sites not shown on the map. The aforementioned study revealed that, as of January 1, 1965, the total area disturbed by surface mining was 3.2 million acres of which 41 percent was from the production of coal, 26.5 percent from the production of sand and gravel, 8 percent from the production of stone, 6 percent each for gold and phosphate, 4-1/2 percent for iron, 3 percent for clay, and 5 percent for all others.

Until the past decade, wastes generated by the materials extraction industry have received little serious industrial attention and almost no public awareness or concern. The practice of dumping mineral extraction wastes at the nearest convenient location, and by the least expensive procedure, has been generally accepted as the inevitable results of exploiting any

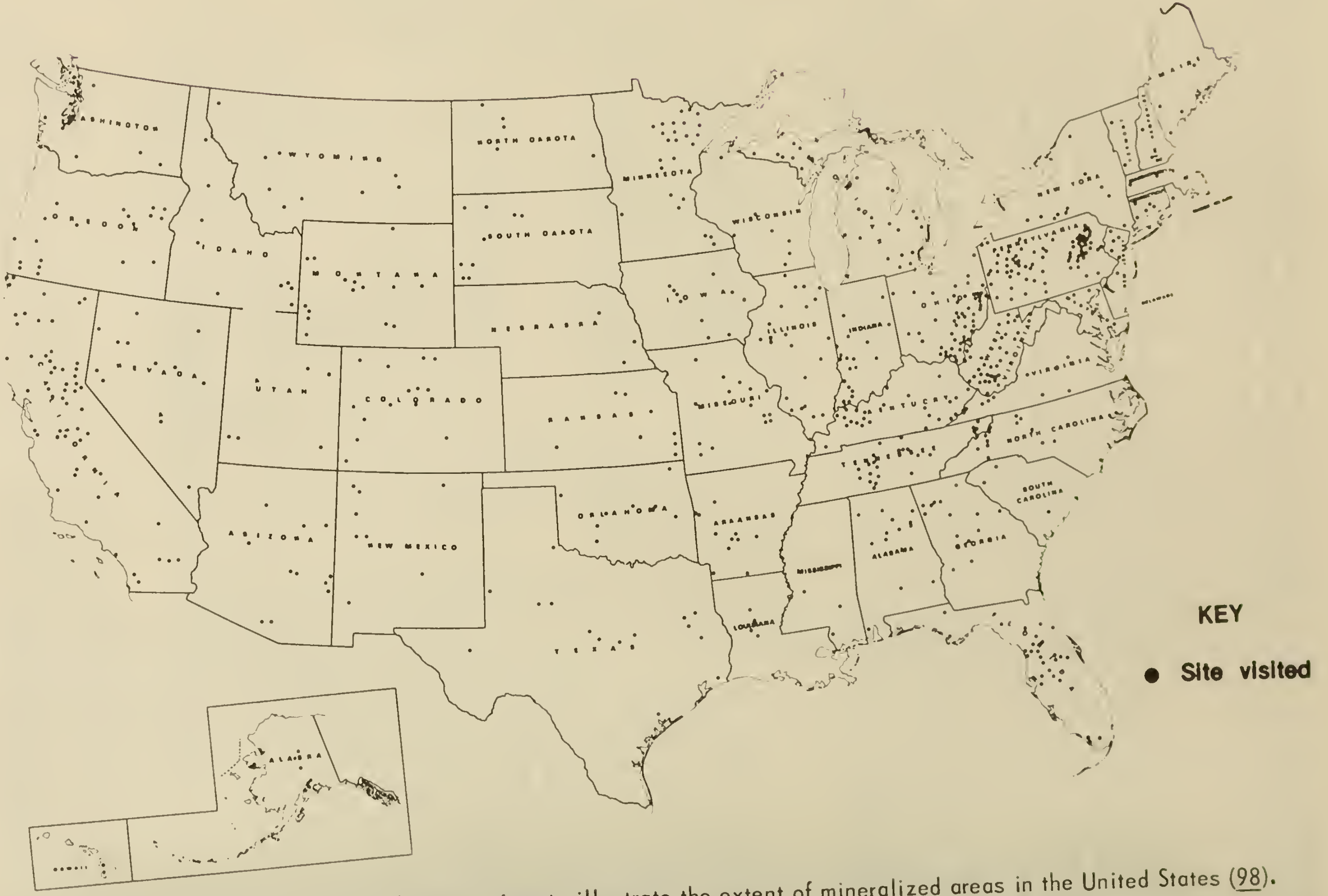

FIGURE 3. - Randomly selected mining sites to illustrate the extent of mineralized areas in the United States (98).

mineral resource. These wastes are still accumulating at accelerated rates, and most of them are still void of any significant value or environmental appeal. Although some waste dumps have been reworked several times through the years as the processing technology has improved, nearly all of the raw materials extraction industry wastes are normally worthless. Immediate low-cost disposal has always been, and continues to be, of primary importance. In the past decade, however, the accumulation of old wastes and the building of new waste sites has been the object of frequent and vigorous charges from environmentalists, ecologists, conservationists, and others who have become increasingly alarmed about current situations, such as the increased incidents of land, air, and water pollution, the impact of wastes on the ecology, and the short supply of many domestic mineral commodities. Bureau of Mines research programs on these and related problems have included investigations on the location, character, quantity, and composition of the waste materials involved, and the development of new processes or techniques that may offer improved extraction, utilization, acceptable waste disposal, and land reclamation technologies.

Recovery and Utilization

As stated previously, the overburden and subgrade rubble wastes from mining and other raw materials extraction operations normally contain no significant amounts of recoverable minerals, nor are they normally useful in significant quantities for any other commercial purpose. However, wastes from several extraction operations such as slate and lime rock quarries, coal mines, phosphate rock mines, and others have been investigated extensively by the Bureau of Mines and in some instances were found to be potentially useful.

Waste Slate for Asphalt Mixtures

One of the first investigations on such recoveries was conducted by Bowles in 1921 (10-11). He studied the use of slate quarrying and cutting dusts for preparing asphalt road surface mixtures, and filler for rubber goods, linoleum, oilcloth, and window shades. He noted that at the time, 80 to 95 percent of the gross slate production in the United States was discarded as waste. It was further noted that the tonnage of filler materials used in asphaltic mixtures for New York City alone exceeded 50,000 tons per year. Of particular interest was the fact that asphaltic mixtures for road surfacing required aggregate materials of sand and stone and fillers with equal amounts of finely pulverized limestone or portland cement. The weight of fillers commonly used with the byproduct asphalts was substantially the same, but less filler was required when Trinidad[3] and certain other asphalts were used.

Waste Slate Dust as Filler

The Bureau of Mines also evaluated the use of slate dust as a filler material in cooperation with manufacturers who were supplied Bureau of Mines sample materials (10) for testing.

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[3] Reference to specific company or trade names does not imply endorsement by the Bureau of Mines.

Bonded briquetes containing either limestone or portland cement filler were found to have no greater impact resistance than comparable briquetes containing slate dust filler. Slate flour was rated between portland cement and crushed limestone when used in standard sheet surface mixtures. The weight of slate filler requirement was equivalent to the limestone filler requirement and about 10 percent less than the optimum amount of portland cement filler. At that time, the cost of slate flour was estimated at about one-half that of portland cement.

The industry also cooperated with the Bureau in an evaluation of slate dust as filler in the manufacture of rubber goods, linoleum, oilcloth, and window shades (11).

Based on reports from the manufacturers who had evaluated Bureau of Mines slate samples in various applications, Bowles concluded that waste slate could be used satisfactorily as filler for some mechanical rubber goods and in some types of oilcloth, floor coverings, and window shade cloth. The most successful tests indicated a need for a uniform slate powder of which 95 percent would pass a 300-mesh screen. If properly pulverized and mixed, the slate dust would be suitable for most rubber goods except those requiring the grade then used in automobile tires. The slate, if properly prepared, could be used to advantage in the production of linoleum, oilcloth, phonograph records, roofing, and many other such products.

Limestone Quarry Waste Recovery

A similar investigation was conducted in 1923 on limestone quarry waste too small in size for use in conventional lime kilns (12). Although no firm data were developed at the time of the report, the author suggested the following recommendations for plant operations and possible uses for the large fraction of small-size limestone generated at the quarry and kiln plant operation.

1. If practical, change lime production circuits to include a mount kiln designed for burning small stone and/or a rotary kiln for burning the fine quarry wastes (less than 2 inches).

2. Use the quarry fines for other purposes such as (a) fluxing stone, (b) roadbed stone, (c) concrete coloring material or aggregate, (d) terrazzo, (e) chicken grit, and (f) soil conditioning.

Gold From Mining Wastes

More recent research (35) was conducted on the recovery of gold from subgrade ore and mine wastes. The materials of interest included the open pit mining wastes generated during the exploitation of low-grade gold deposits, which extend over large areas in northeastern Nevada. Mining wastes going to the dump contained less than 0.05 to 0.08 ounce of gold per ton. An estimated 10 million tons of low-grade ore and mine wastes contained 0.05 to 0.13 ounce of gold per ton. Other known higher grade deposits were too small for exploitation by the conventional countercurrent decantation cyanide process, but both of these resources were thought to be amenable to an economic treatment for gold recovery by dump-leaching techniques developed by the Bureau of Mines.

Following a series of small laboratory tests, Heinen and Porter (35) scaled up their equipment to include leaching columns 27 inches in diameter and closed at the bottom. Test batches were comprised of 200 pounds of waste rock crushed to 1 inch. The leachant contained 2 pounds of sodium cyanide per ton of solution. No lime additions for protective alkalinity were necessary since the water and rock formed a pulp having a pH of 8. The quantity of solution amounted to 400 pounds per ton of rock. An injection system was employed to minimize difficulties normally encountered with clay-bearing wastes.

Test results were highly favorable. At least 80 percent of the total contained gold was recovered after 10 days of leaching. After 21 days, 90 percent of the gold was recovered leaving tails that assayed only 0.006 ounce of gold per ton. Reagent consumption was 0.4 pound of sodium cyanide per ton of mine waste. Part of this consumption was attributed to the adsorption of NaCN by the activated carbon.

The process is now in use by several Nevada gold producers.

Values From Granite-Quarry Wastes

Large amounts of waste materials known as granite fines are produced in quarrying and crushing operations. One granite quarry may produce up to 180,000 tons of granite fines annually. Large amounts of these fines are washed and sold for the production of hot-mix asphalt with little attention given to the possibility of first recovering the contained feldspar and quartz. Eddy, Collins, and Sullivan (26) reported on an investigation of such possibilities in 1974. Their studies were initiated, in part, due to related conservation and environmental problems. They proposed recovering the quartz and feldspar by developing suitable flotation techniques.

The materials investigated were the waste granite fines product obtained from screening a fine-grained biotite granite gneiss mined at Lithonia, Ga. The granite was crushed and screened at 3 mesh, and a 6-ton sample was obtained for investigation from the minus 3-mesh material. Representative samples were obtained for chemical, petrographic, and screen analyses prior to laboratory batch flotation tests.

The principal constituents were 14.1 percent alumina and 73.9 percent silica; other oxides included 4.8 percent K_2O, 3.3 percent Na_2O, 2.3 percent Fe_2O_3, and substantially lesser amounts of MgO and other minerals. The samples also contained about 0.2 percent combustible material. The three principal mineral constituents included 31 percent quartz, 30 percent microcline, and 26 percent plagioclase; the lesser minerals included 9 percent biotite, 2 percent muscovite, and 2 percent of all others.

Laboratory flotation tests were designed for the separation of biotite, feldspar, and quartz. Flotation variables included (1) the effects of pH on biotite removal in acid and alkaline circuits, and (2) the reagent schedule for selective removal of soda feldspar and recovery of potash feldspar. Ultimately, flotation systems were developed for continuous flotation recovery of the feldspar and quartz.

Laboratory batch tests demonstrated the feasibility of recovering high-grade quartz and feldspar products and the removal of biotite by either sequential froth circuits or by one flotation circuit and supplemental wet-magnetic separation. The high-grade feldspathic sand product was obtained by removing the fine-size biotite in an alkaline circuit and the coarse-size biotite in an acid circuit.

The biotite product contained 19.3 percent Fe_2O_3 and represented more than 71 percent of the iron in the fine wastes. Good froth flotation was achieved by removing primary and secondary slimes at 400 mesh.

Reclamation, Stabilization, and Disposal

In a 1972-73 survey (62) of land utilization and reclamation in the mining industry for the period 1930 to 1971 inclusive, it was determined that the land utilized by the mining industry totaled 3.65 million acres or 0.16 percent of the total U.S. land mass. The term "reclaimed" was considered to mean that all restoration or reconditioning accounted for during the survey had been completed in compliance with Federal, State, and local laws in force at the respective locations. It also includes areas where no reclamation was required by law but restoration had been completed and judged to meet normal standard specifications then prevailing at other areas. In addition, it presumed that minimal environmental pollution would occur at the restored sites.

The amount of land reclaimed from 1930 to 1971 was 1.46 million acres or 40 percent of the total land disturbed. In 1971, 206,000 acres were disturbed, but 163,000 acres (79 percent) were reclaimed. This was nearly double the percentage reclaimed in the previous 40 years. The mined commodities for which these lands were disturbed and the respective percentages of the total land utilized by the mining industry were as follows: bituminous coal, 40 percent; sand and gravel, 18 percent; stone, 14 percent; clay and copper, 5 percent each; iron ore, 3 percent; and phosphate rock, 2 percent. Land disturbed in the extraction of all other mineral-based commodities totaled 13 percent.

The fossil fuels and nonmetals extraction industries each accounted for 43 percent of the total land utilized (1930-71); the metal mining industry accounted for the remaining 14 percent.

Over 1 million acres of the reclaimed land was by the fossil fuels industry or 69 percent of the total; the nonmetals industry reclaimed 28 percent of the total, and the metals mining industry reclaimed 3 percent. About one-half of the total reclaimed acreage was attributed to the efforts of the bituminous coal industry.

Revegetation of Strip Mine Wastes by Fertilizer and Lime Additions

Research on the reclamation of strip mine waste areas by vegetation cover (45) was conducted to develop data on the appropriate ground preparation and types of vegetation suitable for typical backfilled areas in north-central Pennsylvania. The Bureau of Mines investigation included complete backfilling or partially backfilled sites, revegetation of strip mine spoil, and acid mine drainage. The studies were conducted with the support of a committee comprised of members representing the Bureau, the Soil Conservation Services, the Forest Service of the Department of Mines and Mineral Industries, the Department of Forests and Waters, and the Game Commission of the Pennsylvania Commonwealth.

After proper backfilling and soil preparation, the soil conditions were analyzed in order to estimate essential additions of lime and fertilizer. The committee prescribed the test conditions and the selection of plants and seeds for the initial planting.

It was found that the resulting ground cover of grass-legume mixture increased markedly with increasing soil pH levels up to 5.5. Increasing the pH to 6.5 produced no appreciable additional ground cover.

It was noted that areas fertilized (500 pounds per acre) and limed to pH 5.5 produced no better ground cover than an unfertilized area of the same pH. It also appeared that seed mixtures containing some types of fescue produced a better ground cover than mixtures having no fescue. Mixtures containing creeping red fescue and sericea lespedeza produced the best cover, averaging 45 percent of the area seeded.

A study of the survival rate of the planted trees indicated that Norway spruce was the best suited for the area. Of the 220 spruce trees planted, 82 percent survived. More than 70 percent of the four other species also survived. They included black locust, Japanese larch, and pitch pine. The survival rate of four other species exceeded 50 percent. These included jack pine, lespedeza natob, bristly locust, and autumn olive.

Reclamation of Acidic Coal Mine Spoil by Fly-Ash Additions

The acidic nature of coal mine spoil at many mine sites precludes natural revegetation of lands disturbed by surface mining operations. The foregoing discussions pertain to the use of lime to increase the backfilled soil pH sufficiently to support covering plant growth. However, the basic chemical nature of industrial fly ash suggested an extensive study of fly ash as potentially effective and much cheaper than lime (1). In 1965, the Bureau of Mines, in cooperation with the West Virginia University Agronomy Department and the Soil Conservation Service of the U.S. Department of Agriculture, conducted an extensive investigation on the use of fly ash for mine spoil reclamation. The objectives were to (1) determine if fly ash would increase the soil pH sufficiently to support vegetative ground cover, including grasses and legumes, (2) find the optimum ratio of fly ash to spoil, and (3) determine whether treated spoils would maintain vegetation long enough to increase organic matter in the soil and thus reduce the toxic potential of the ash. Since the total U.S. production of industrial fly ash from electric generating stations amounted to more than 30 million tons annually in 1965, an underlying objective was to find an economic outlet for large tonnages of fly-ash waste.

The experiments demonstrated a firm potential for the application of large amounts of fly ash for the successful reclamation of mine-spoil areas. The fly ash was found to effectively neutralize the highly acidic spoil and produce mixtures that were satisfactory for seed generation and plant growth. The plant species that thrived on ash-spoil soil included Kentucky 31 fescue, orchard grass, rye grass, redtop grass, and birdsfoot trefoil. The grass yield was equal to or better than the average for the State of West Virginia.

Large amounts of fly ash were required to raise the pH to satisfactory levels, and in some areas the amount of ash was sufficient to change the texture of the spoil. This condition also appeared to improve the microbiological character or the spoil-ash mixtures. Nodules of active nitrogen-fixing bacteria were found on the roots of birdsfoot trefoil, thus indicating a pH level suitable to support these important micro-organisms.

Extrapolation of recorded data indicated a lapse of about 20 years for the pH levels of control plots to rise from 2.5 to 5.0. Similar extrapolations for ash-treated plots indicated that from 1 to 10 years would pass before the pH would drop from levels of 5.3 and 8.7 to a level of 5.

Figures 4 and 5 show the contrast of vegetation cover for treated and untreated spoil.

Subsidence Control by Pumped-Slurry Backfilling With Wastes

The Bureau of Mines interest in hydraulic backfilling dates back to the first year of its existence in 1910. The First Annual Report of the Director, J. A. Holmes, in 1911, describes research on hydraulic backfilling as a potential method to reduce the annual waste of anthracite, make coal mines safer for the workers, and reduce settlement of the surface (38). An intensive interest in hydraulic backfilling was revived in 1972 when the Bureau of Mines, in cooperation with others, conducted studies and demonstrations (19) on hydraulic backfilling technology as a disposal technique applicable to large accumulations of subsurface mining wastes. Other objectives were to develop new solutions to the problems of surface subsidence at worked-out and abandoned locations; reduce surface solid waste polution, and extinguish waste-bank fires. The technique of interest included the design and construction of a system to slurry crushed mine waste and transfer the slurry into mining voids so as to distribute it throughout extensive mineworking voids from a minimum number of borehole supply lines.

Full-scale demonstrations at mines in Scranton, Pa., and Rock Springs, Wyo., were completed in 1973 (100). The sites were selected because these

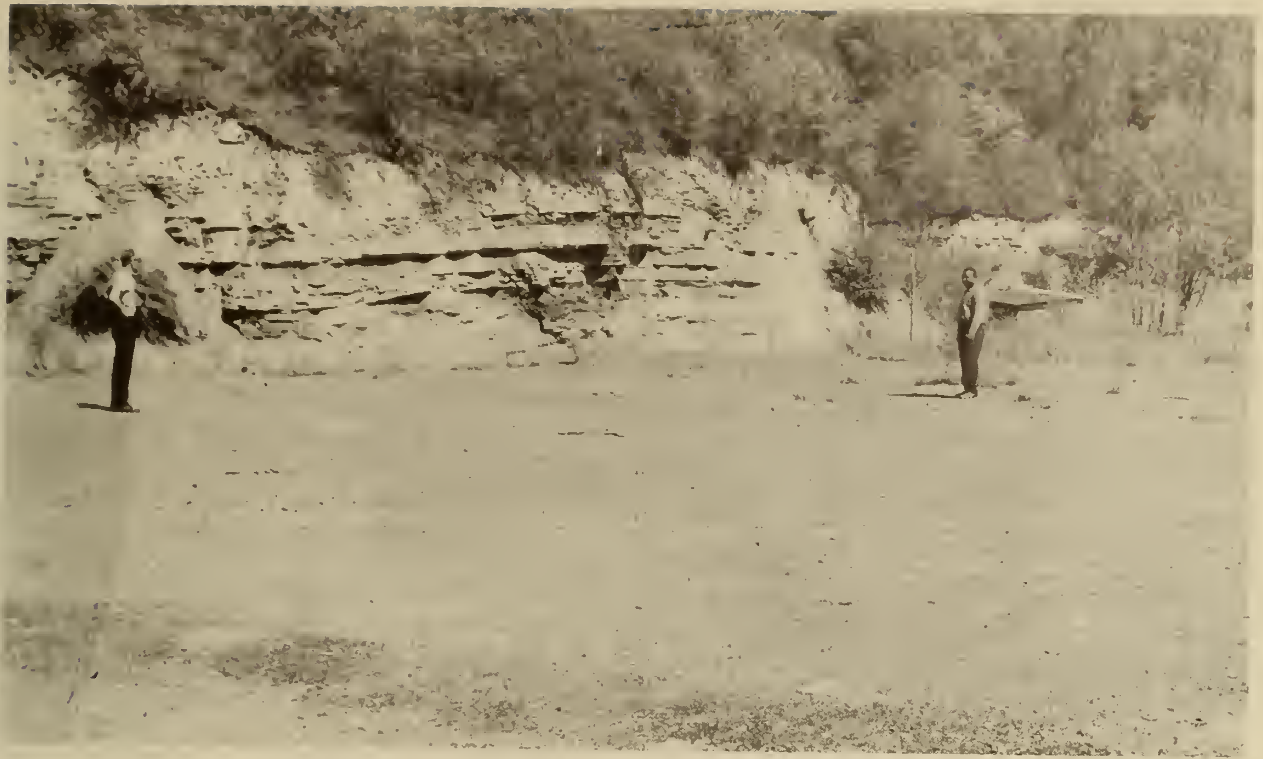

FIGURE 4. - Vegetation on untreated mine spoil (2).

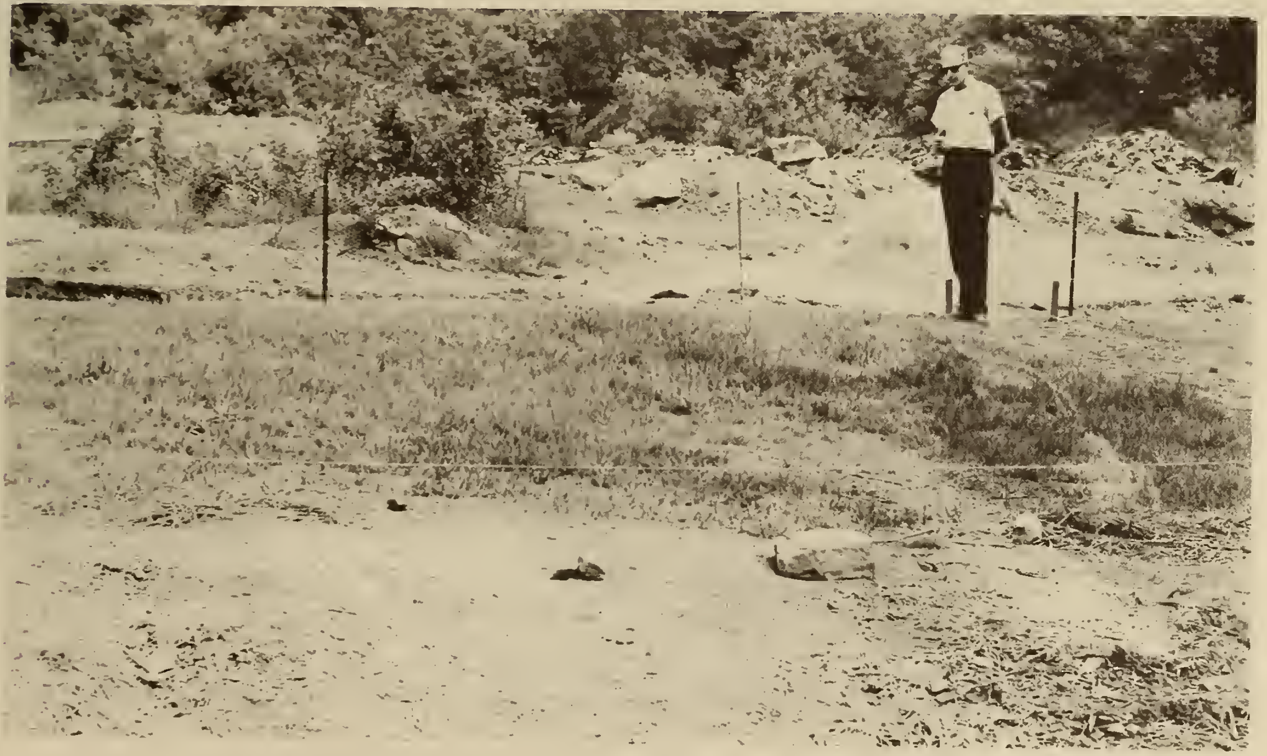

FIGURE 5. - Vegetation on a portion of treated mine spoil (background) and untreated area (foreground) (2).

communities were underlain by several mined-out coalbed layers which left extensive inaccessible voids into which subsidence had extended to the surface at several locations. The backfill operations were performed by the Dowell Division, Dow Chemical Co., Houston, Tex. The Bureau of Mines cooperated in the Rock Springs, Wyo., demonstration and conducted the Scranton demonstration under contract.

After many tests with slurry transport systems and mine models, the demonstration sites were selected and full-scale systems were designed.

The two types of backfilling techniques considered were known as controlled flushing and blind flushing. Although slurries can be injected either by gravity flow or pumping, the latter has the definite advantage of distributing the slurry far more extensively than can be accomplished by gravity flow.

Controlled hydraulic backfilling can be performed when the mine voids are still accessible for workmen to freely place the slurry transfer system. Blind hydraulic backfilling must be employed when the mined-out area is no longer accessible and the slurry must be delivered from the surface into the abandoned voids through borehole pipes. Pumped-slurry blind flushing was found to have the following advantages over the gravity feed technique: (1) Fewer injection boreholes were required, (2) the vertical filling or voids was more complex, (3) mine areas, inaccessible because of surface structures, could be filled with little or no surface disturbances, and (4) less disturbance of the community.

In the Scranton demonstration, a residential area of 30 acres was stabilized by injecting 450,000 cubic yards of crushed mine waste into the two mining voids through only five injection boreholes. In one system configuration having a single centrally located delivery hole, 200,000 cubic yards of waste was injected and transferred to all sides of the working cavities, reaching a maximum lateral distance of 640 feet from the injection pipe. The cavities were filled from floor to roof. With the formerly used gravity method, the average amount of injected waste per hole was only 300 cubic yards. In general, backfilling by gravity feed required many closely spaced injection holes and the backfill was never complete. Figure 6 is a view of the Green Ridge section of the Scranton demonstration site. The dashed line encloses the backfilled area, which has been stabilized and no longer in danger of sinking caused by mine subsidence. Figure 7 shows the mixing tank, water and slurry pipes, the pump, and the instrumentation and control equipment trailer.

Mine Water Neutralization and Other Processing Techniques

Many U.S. mines are cut into rock strata that have been penetrated by natural water channels, which often release large volumes of water into the mine workings. In some cases, the water leaves the mine by gravity flow to a surface opening or through outflowing seepage channels. More frequently, it is collected in water sump areas in the mine and pumped to surface discharge locations. In many mineralized areas, such as the Appalachian region,

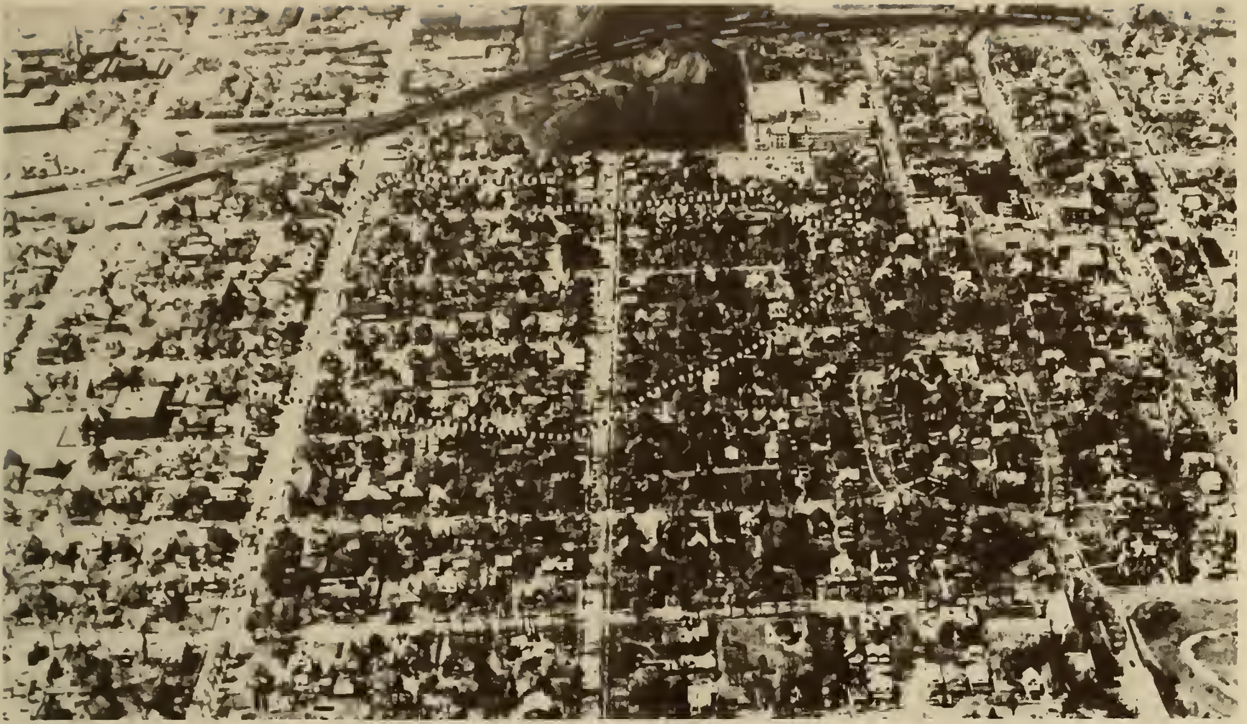

FIGURE 6. - Site of backfilling demonstration, Green Ridge, Scranton, Pa. (dashed line encloses backfilled area) (95-96).

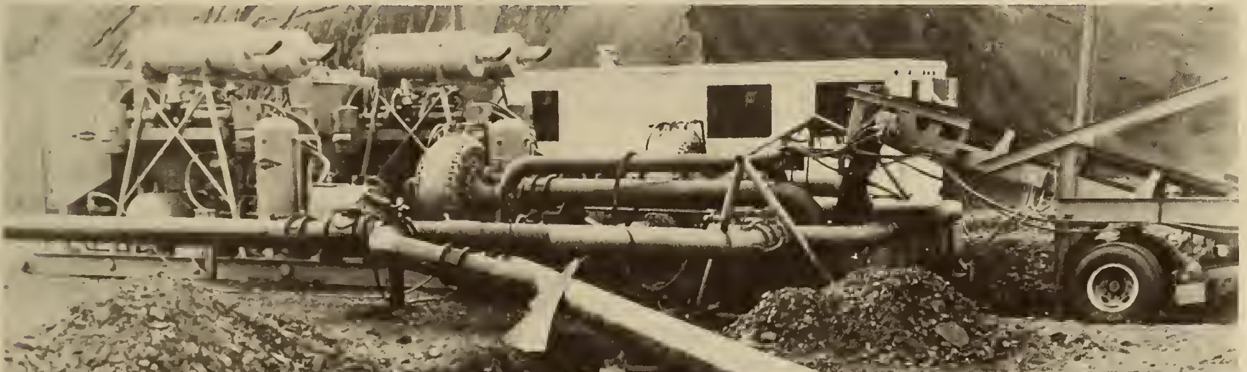

FIGURE 7. - Slurry-mixing and pumping equipment for forced backfilling (mine spoil in background) (95-96).

the inflowing water reacts with various exposed pyritic and other constituents of the mineral beds and/or the overburden (25). Eventually, it becomes polluted with sulfuric acid and dissolved or suspended solids. It is then known as "acid mine water," which is characterized by a low pH and relatively high concentrations of metallic salts, primarily iron and lesser amounts of metals such as aluminum, magnesium, and calcium. Typically, the total acidity is comprised of free sulfuric acid and the mineral acidity generated by the hydrolysis of iron, aluminum, and other metal salts.

The Bureau has investigated a number of techniques to prevent acid mine water pollution by neutralization, or by preventing or slowing the acidification process.

Limestone Neutralization

Proper treatment of acid mine water requires oxidation of the soluble ferrous iron to insoluble ferric iron, which will settle out with other suspended solids. The sulfuric acid must be neutralized with lime, limestone, or some other chemically basic material such as industrial fly ash. During 1966 and 1967, the Bureau of Mines conducted research on the chemistry of acid mine water, the use of limestone as a neutralizing agent, and ferrous iron oxidation (25, 51). Investigations indicated that oxidation of ferrous iron could be accomplished by several means, and limestone could be equally as effective as lime for neutralization and probably much cheaper. In 1967, Mihok, Duel, Chamberlain, and Selmeczi demonstrated a large-scale processing concept at a mine site (52). Water treatment was completed in four steps as follows: (1) Preparing a limestone slurry with limestone pulverized in an autogenous mill to minus 400 mesh, (2) mixing the lime slurry with mine water, (3) aerating mixed slurry and mine water to oxidize and precipitate iron, and (4) settling the solids and draining the neutralized water.

The mine water at the demonstration site had a pH of 2.8. It contained 1,200 ppm total acidity, 36 ppm ferrous iron, and 360 ppm total iron, and it was treated at rates of 300 to 400 gpm. The treated effluent water was discharged at a pH of 7.4 and contained no detectable iron. The volume of compacted sludge, with limestone neutralization, was about one-third of the amount removed when neutralizing with lime. The system is illustrated in figure 8. The 3-million-gallon holding pond was employed to level out the pH variations of the mine discharge water. The autogenous tube mill effectively ground 3-inch limestone to less than 400 mesh. Ferrous iron was oxidized by blowing air, at 100 cfm, through an aerator operating in 60,000 gallons of a limestone slurry-acid water mixture. The effluent from the aerator pond had a pH of 6.8 and 3 ppm of iron. Water discharged from the settling pond had a pH of 7.0 and less than 1 ppm of iron.

In another more specific investigation, Mihok (50) found that ferrous iron in highly ferruginous acid mine water could be rapidly oxidized with activated carbon, indicating that the air oxidation is catalyzed by the activated carbon. It was necessary to condition the activated carbon repeatedly with the acid mine water and air. The reaction was found to proceed rapidly in an acid environment of pH 2.5 engendered by the oxidation

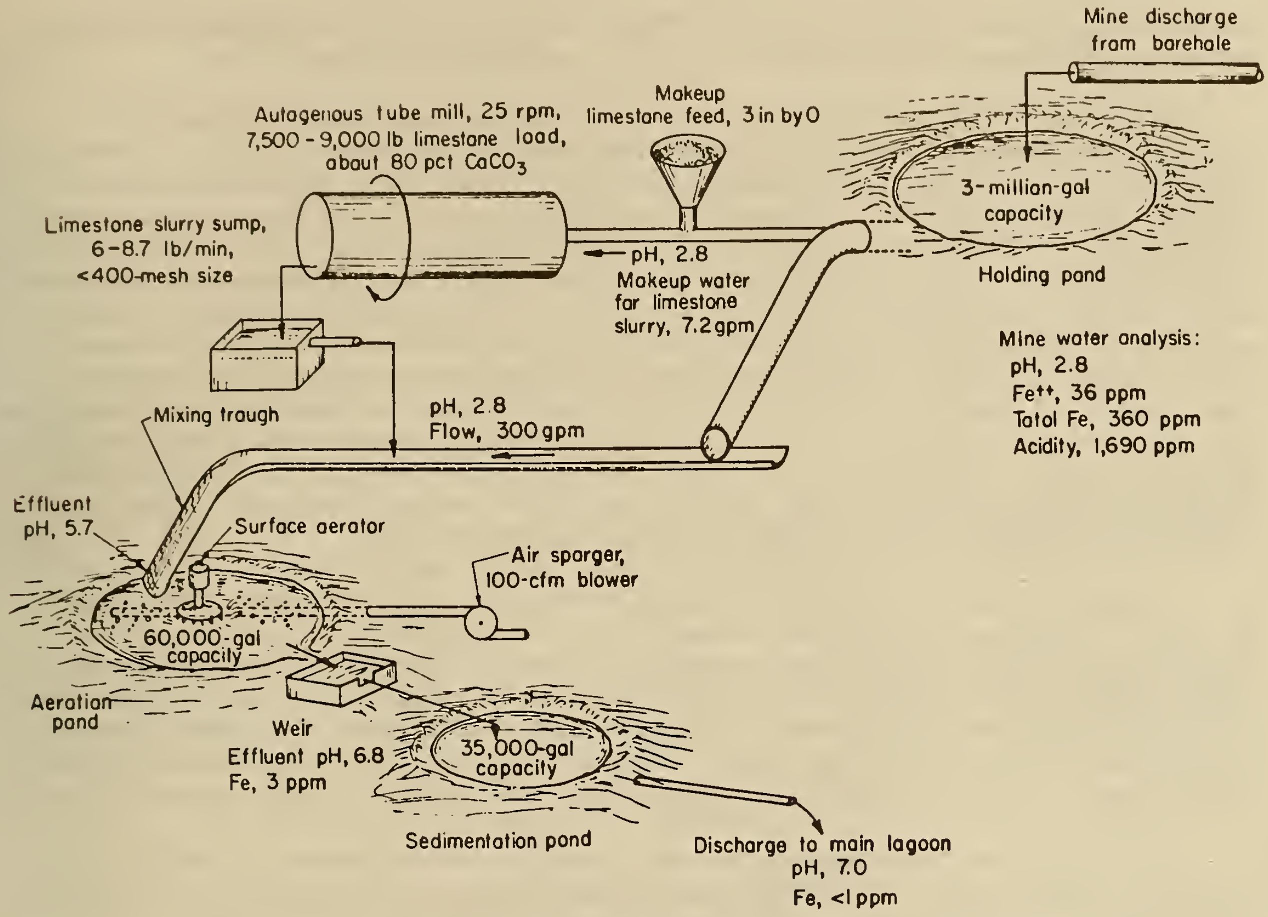

FIGURE 8. - Limestone neutralization of acid mine water (52).

of ferrous iron. The carbon retained its efficiency after repeated usage with no discoloration or deposition of solids. Mihok concluded the the catalytic oxidation of ferrous iron would offer a more precise pH control, lower reagent cost, preclude the need for aeration systems, and permit smaller holding ponds.

Recovery of Uranium

The water effluent from some mines contains valuable and recoverable constituents. The acidic water from some copper mines and waste dumps, for example, also contains dissolved copper, which is commonly recovered by a cementation process in which iron (from scrap) replaces the copper in solution. Copper is collected as a settled sludge in the processing vats, and the dissolved scrap iron is carried away with the effluent water.

In other areas such as the Ambrosia Lake District of New Mexico, the mine water is slightly alkaline and contains uranium as the $UO_2(CO_3)^{-4}$ complex. In a year of investigations (1968-69), Ross and George demonstrated a countercurrent ion-exchange process for absorption and elution of uranium from the

Ambrosia Lake mine waters (70). A demonstration was conducted with the cooperation of Kerr-McGee Oil Industries, Inc., and United Nuclear Corp.

More than 5 million gallons of mine water was processed through a multiple-compartment exchange column, 14 inches in diameter. Bureau of Mines participation included the designing and constructing the equipment, supervising the installation, training operating personnel, and periodically collecting and reviewing operating data. Since pilot-plant elution equipment was available at the Bureau's Salt Lake City (Utah) Metallurgy Research Center, tests on the continuous elution or stripping of uranium from loaded resin was made using loaded ion-exchange resin from the United Nuclear Corp.'s mine water treatment plant. Ross and George describe the equipment, techniques, and results of field and pilot plant tests including information on the design and hydraulic performance of a large multiple-compartment ion-exchange column. The mine water, containing 9 to 12 ppm of U_3O_8, was treated in an absorption column comprising five or six compartments, each 4 feet in depth. Each cubic foot of the resin load absorbed up to 3.6 pounds of U_3O_8.

Uranium recovery efficiency averaged 98 percent at solution flow rates of 15 to 25 gallons per minute through the 4-inch-diameter elution column. The elution test equipment was comprised of the 4-inch-diameter countercurrent column and a 9-foot bed of loaded resin. Resin loaded to the 3.6 pounds of U_3O_8 per cubic foot was eluted to a residual of 0.06 pound per cubic foot with a 224-minute resin-retention time. Only 1.9 volumes of stripping solution containing 1.1 $\underline{M}$ NaCl and 0.5 $\underline{M}$ $NaHCO_3$ were required for each volume of resin.

This improved ion-exchange recovery method was estimated to reduce conventional ion-exchange equipment requirements then in existence by at least 70 percent. A substantial reduction in capital and operating costs could also be expected.

The technique is now a relatively standard procedure in the uranium producing industry.

Air-Sealing of Coal Mines To Reduce Water Pollution

It has long been known that the rate of pyrite oxidation and resulting sulfuric acid formation is a function of the oxygen concentration in the presence of the contacting water. It has also been noted that some strains of bacteria can produce a catalytic effect in the oxidation process (42). It has been reasoned, therefore, that acid mine waters could be prevented from forming if the pyrite in the local rock strata and coalbeds could be protected against oxidation by techniques such as sealing the mines to keep the air out. Such techniques have, in fact, been studied by various groups including State and Federal agencies since 1923, but results were not totally conclusive.

In 1964, Moebs and Krickovic (53) began a new extensive investigation of the air-sealing concept at carefully selected mine sites in order to obtain additional firm data. Their objective was to test the possibility of sealing the mine and measuring the resulting change in the acidity of the effluent water.

The 77-acre coal mine demonstration site was situated above the level of natural water drainage. The mine waters were monitored at the site for more than 2-1/2 years prior to air sealing in 1966. The mine air and water were then monitored for an equal period to determine the exact trend of oxygen depletion in the mine atmosphere after sealing and the resulting change in the effluent water acidity. The air samples were taken through the seals and one borehole drilled from the surface. Water was allowed to flow by gravity out of the mine through special water seals at several locations. These seals kept outside air from entering the mine but provided for the free flow of effluent. All other openings to the mine were sealed with impermeable structures or materials.

Prior to sealing, the average total acidity was observed to decrease from about 620 ppm in mid-1964 to about 410 ppm 2 years later. Immediately after sealing in 1966, the average total acidity dropped to near 280 ppm and gradually decreased to about 180 ppm during 2 additional years.

In some cases, air sealing may also reduce the inflow of water and thus lower the rate of waste-water discharge and hence the acid load; however, no pronounced change in water flow could be attributed to air sealing in this investigation.

RAW MATERIALS BENEFICIATION

Recovery and Utilization

The United States used more than 4 billion tons of new mineral supplies in 1974 (97) or about 40,000 pounds for each of its citizens. This included 20,750 pounds of nonmetal minerals such as salt, clay, cement, sand and gravel, stone, and others; 1,455 pounds of metals such as iron and steel, aluminum, copper, zinc, lead, and others; and 1,785 pounds of energy materials such as petroleum, coal, natural gas, and uranium. The energy materials alone were enough to supply each citizen with energy equal to that produced by 300 persons working 24-hour days for a full year.

The raw material products of the minerals extraction sector, previously described, are taken as crude materials into the raw-materials beneficiation sector where they are converted into specification-quality products, which supply the subsequent bulk raw materials processing sector. In effect, the beneficiation industries function to separate and recover the wanted from the unwanted constituents of the incoming raw materials. The net effect is a transfer of values from the incoming resource materials to the outgoing products with a concurrent generation of wastes. Since the weight of these wastes comprise a very large fraction of the incoming raw materials, the weight and volume of wastes generated in the raw materials beneficiation industries are usually many times greater than those of its products. Most of these wastes have been accumulating for years at the industries' disposal sites.

Recovering Copper From Mill Tailings

The well-established flotation process is one of the most commonly used methods for the separation and recovery of metal-bearing minerals from the nonmetal host constituents in all ores. However, since very few, if any, minerals beneficiation processes now in commercial use are 100 percent efficient, some of the desired constituents are always lost in the tailings discharged from flotation plants. Because of the extremely large tonnage of mineral concentrates produced annually, only a slightly higher beneficiation efficiency would increase the total metal production significantly. Although the tailings normally retain a very small percentage of the desired minerals, there has been a continuing search for improved flotation techniques and other concentration processes to reduce tailings losses.

During the late 1960's and early 1970's, White, Fergus, and Goff conducted a series of selected studies on the recovery of copper from mill tailings by flotation (103). The general objective was to improve the current flotation practice and explore the potential for extracting the low levels of copper minerals contained in the millions of tons of available copper flotation mill tailings. More specifically, the objectives were (1) to determine the grade of concentrates and recovery obtainable by scavenger flotation techniques, (2) to determine the results of regrinding and cleaner flotation of the scavenger concentrates, and (3) to determine the amounts of copper that could be recovered from tailings by leaching with cyanide and comparing the results with flotation data. All tailings used in the test series were contributed by 11 western U.S. copper mills.

The studies revealed that although all tailings do not respond identically to any specific scheme of beneficiation, comparisons and generalizations among the different test samples could be made. Some of the conclusions follow:

1. The amount of sulfide copper minerals initially in the tailings was the most significant variable controlling flotation concentration. An empirical equation could be developed for determining the amount of "unrecoverable copper" in the tailings.

2. The tailings from cyanide leaching were generally lower in oxide copper minerals and higher in sulfide copper minerals than the tailings from rougher flotation.

3. The major problem in recovering copper minerals from existing tailings is the locking of sulfide minerals with the gangue (waste rock) materials. Since most of the copper minerals in the test tailings were locked in the particles of greatest diameter, additional recovery could be achieved by successive steps of additional grinding and subsequent flotation.

Leaching Gold-Bearing Mill Tailings

Beneficiation mill wastes are generated in many different ways and types. The aforementioned flotation tailing is but a single type. Different tailings are generated in leaching processes such as the cyanide recovery of gold from natural or pretreated ores. Because gold is such a high-value commodity, there is often a greater economic incentive to process wastes containing gold than similar wastes containing other lower value metals such as iron, copper, and lead.

In 1970, Nichols and Peterson (58) reported on their investigation of potentially profitable methods for re-treating the gold-bearing mill tailings left at the former Mercur, Utah, gold-producing operations.

Over 5.5 million tons of ore, containing 920,000 ounces of gold, was processed in the Mercur District between 1871 and 1917. An estimated 750,000 tons or ore, containing more than $3 million in gold, was processed between 1934 and 1942, at which time mining operations ceased. It was estimated that the tailings dumps at Mercur, Utah, contained about 0.07 ounce of gold per ton, worth about $7 million. The research by Nichols and Peterson was aimed at evaluating current technology for reprocessing the valuable Mercur tailings. The sampling work, begun in 1966, comprised auger drilling at selected representative sites and subsequent blending of selected sections of the augered materials.

Reprocessing experiments were conducted after careful mineralogical and metallurgical studies. The experimental reprocessing test included (1) water and cyanide leaching, (2) cyanide leaching modified by pretreatments with oxidants, acids, bases, and salt water solutions, (3) a combination flotation-cyanidation, and (4) roast-cyanidation.

Although an economical gold-recovery process was not found, the following firm conclusions were developed:

1. Simple water leaching recovered 10 percent of the gold.

2. Cyanide leaching at 70° C for 24 hours recovered 42 percent of the gold including the water-soluble gold.

3. Pretreatment with hot sulfuric acid followed by cyanide leaching recovered 49 percent of the gold.

4. Addition of 120 pounds of sodium hydroxide per ton of tailings to a 24-hour, 70° C cyanide leach improved gold extraction to 60 percent.

5. Preroasting at 530° C for 4 hours followed by cyanidation of the roasted tailings gave a 65-percent recovery.

6. Leaching the above roasted tailings for 6 hours in 90° C sulfuric acid followed by lime neutralization and cyanidation gave gold recoveries of 79 percent.

Utilization of Copper Mill Tailings for Building Bricks

The major copper-producing mines in the United States are located in the intermountain States of Utah, New Mexico, Nevada, Montana, and Arizona. The ores from these mines average less than 1 percent copper, which means that enormous amounts of tailings are generated by the flotation mills in order to recover acceptable smelter-grade concentrates. The estimated annual production of tailings at these mills exceeded 125 million tons in 1971. At annual rates of this magnitude, the total accumulation of copper mill tailings has caused considerable concern in some quarters during the past decade. Such concern has prompted an extensive search for potential commercial utilization of at least a part of the total accumulated tailings.

In 1971, Pigott, Valdez, and Dean (63) reported on the use of copper mill tailings for making dry-pressed building bricks.

In preliminary materials studies, they determined that the principal mineral constituents of five tailings samples ranged from 66.4 to 74.6 percent SiO_2; 6.2 to 13.2 percent Al_2O_3; 2.6 to 11.4 percent FeO; 2.1 to 6.0 percent K_2O; and lesser amounts of calcium, magnesium, and sodium oxides. The principal minerals included quartz, feldspar, biotite, sericite, and muscovite. Minor constituents included pyrite, chlorite, illite, andalusite, amphibole, apatite, and hematite.

The evaluation of these materials for making building brick by the dry-pressing method included drying, screening, mixing with color and binding additives, adding water, forming, firing, and cooling. The additives included varying amounts of calcium, lignosulfonate, manganese dioxide, and dolomite. Firing temperature ranged from 1,120° to 1,230° C. The forming pressure was 5,000 pounds per square inch.

Resulting laboratory bricks were equal to or better than ASTM specifications for solid facing brick (FBS). The encouraging laboratory results prompted a pilot-plant-scale investigation of the process and materials at the Coal Research Bureau, West Virginia University, Morgantown, W. Va. Two different mixtures were prepared from a 5-ton shipment of tailings. Two different mixtures were used to produce standard-size bricks. One contained 99.5 percent tailings and the other 99.0 percent. The balance in each mixture was calcium lignosulfonate. Dry-pressed bricks fired at 1,150° C for 9 hours developed an attractive salmon color and excellent physical properties, considering the relatively short firing period. The pilot-plant bricks like the laboratory bricks met ASTM specifications C62-66 for severe weathering (SW) and moderate weathering (MW) grades of the FBS type.

In a subsequent economic evaluation of the process, it was found that production costs for a plant producing 22.2 million bricks annually would be about $34 per 1,000 bricks having a postulated market value of $80. Although this indicates an attractive return on the investment, the freight rates for brick and/or tailings could alter the overall economics significantly. It was estimated that shipments of brick from Salt Lake City to Denver and San

Francisco would increase the cost of delivered brick to $62.99 and $64.19 per 1,000, respectively.

Dewatering and Densification

Three-fourths of the U.S. phosphate rock production comes from Florida phosphate operations. Unfortunately, the wastes generated in the beneficiation of phosphate rock comprise a clay-slime slurry of about 5 percent solids (92). Waste disposal problems are compounded by the fact that the volume of waste slime generated is 1.5 times greater than the volume occupied by the rock before it is mined. The disposal practice has been to impound the slimes behind high dykes or dams where a slow settling process continues for more than a decade. Shortly after impoundment, the slime acquires the consistency of chocolate pudding and subsequent settling and drying are extremely slow.

Electrophoresis and Coagulation of Phosphate Slime

The slime disposal problem continues to be aggrevated by the high cost of disposal sites and the steady decrease of available disposal areas. These and other problems prompted a cooperative effort by the Bureau of Mines and the Florida Phosphate Council (representatives of the mining companies) to find ways to coagulate the suspended solids in slimes and precipitate them in the shortest time possible. Terichow and May (92) conducted an investigation of several methods to promote rapid settling of solids by the addition of other particulates systems not intrinsically associated with the slime constituents. Many previous investigators had studied the typical characteristics of slime which control settling and drying. Several complicated theories have evolved to explain the particle behavioral patterns observed in such solutions. Most of these theories involved the solid particle-liquid interface layers caused by electrical-charge forces between liquid and solid surfaces, and the attending ionic layers on and near the particle surfaces. The major controlling factors were observed to be directly related to the average mobility of the suspended particles. The research approach was thus aimed at finding ways to disrupt the rather stable colloidal systems by causing mutually attractive forces between the slime particles, possibly with others that might be introduced. Ultimately, the tests included the addition of silica sand and gypsum. These tests were compared with others in which the same concentrations of aluminum sulfate were tested under identical conditions. Electrophoretic mobility measurements were based on the standard direct measurement of the movement of visible microscopic particles observed between the platinum electrodes of a test cell. The additives studied included commercial Ottawa sand (minus 200 mesh), commercial gypsum, and reagent-grade aluminum sulfate $Al_2(SO_4)_3$.

Experimental results indicated that 0.03 N aluminum sulfate solution would produce mutual attraction between colloidal particles of slime and Ottawa sand, thus promoting slime dewatering by flocculating the suspended solids. An economic evaluation of such a system applied on a commercial scale was not made.

Electrodewatering Phosphate Slime

About 8 billion gallons of water is trapped annually in the slimes from phosphate rock beneficiation plants. Not only do the slimes present a disposal problem, they also, in effect, tie up billions of gallons of water that could otherwise be reused repeatedly at the washer plants, thus reducing the large volumes lost at the disposal sites.

In 1963, Stanczyk and Feld (88) reported on the slime dewatering work performed cooperatively by the Bureau of Mines and the University of Alabama in 1961-63. This study of electroosmotic dewatering of slimes was prompted by many observations of this and related phenomena by previous investigators beginning about 1807 and continuing to the present. Although considerable data and sophisticated theories have been developed (56, 59, 93), there have been relatively few commercial applications of these phenomena for concentrating colloidal solids in solution. In simplest terms, electrodewatering occurs in some slurry systems when an electrical potential is applied across a portion of the slurry causing either the solid particles or the water to migrate to one of the electrodes leaving relatively dewatered solids at one electrode and clear water at the other.

Slimes from two sources were studied. One from a 1-year-old settling pond contained about 14 percent solids. The other of undetermined age contained about 17 percent solids. The first sample was used for small-scale dewatering tests with charges of up to 500 pounds. The other sample was used for large-scale test charges of various weights up to 4 tons.

The principal constituents of both samples were 32 to 33 percent SiO_2, 16 to 18 percent CaO, 15 to 16 percent Al_2O_3, and 11 to 12 percent P_2O_5. Organic material amounted to 13 to 15 percent. Petrographic examination revealed the presence of quartz, attapulgite, kaolinite, collophane, and other minerals of the apatite group.

The laboratory test cell was a hollow, 1-foot, cube-shaped, open-topped vessel with 4-1/2-inch cylindrical anodes placed vertically at the four corners. The cathode was a large-diameter cylinder made up of 4-mesh iron wire with a solid bottom and covered with two layers of cheesecloth. A 3-inch cylindrical cathode was placed concentrically within the wire mesh cylinder. The capacity of the 1-foot cell was about 50 pounds of slime. A much larger cell of box configuration with various electrodes in a number of configurations was used with slime batches of up to 4 tons.

Results showed that dewatering by this technique could achieve up to 35 percent solids but the process was slow. The rate of dewatering was found to be essentially independent of cathode surface area.

The combination of electrodewatering and vacuum filtering 15 percent slimes to 35 percent solids decreased the time by 25 to 40 percent. It was also found that electrical power requirements for rapid dewatering to acceptable limits was much too high for economic commercial application of the process.

Flotation of Tennessee Phosphates From Beneficiation Wastes

In 1966, Lamont, Spruiell, Brooks, and Feld (40) conducted a series of experiments designed to recover phosphates from the wastes generated during previous but inefficient beneficiation operations in Tennessee. Anionic flotation concentration had been employed during the 1930's to produce superphosphate and wet-process phosphoric acid. Although the phosphate concentrates contained 36 percent P_2O_5, the loss of P_2O_5 in the tailings was very high by present standards. The total losses in washer and flotation operations often exceeded 50 percent of the phosphates mined.

The aim of the 1966 investigations was to study various techniques for treating the several hundred thousand tons of wastes generated during the early operations, and to improve the grade of product and process efficiency of current beneficiation practice.

Initial phosphate flotation tests were made with the waste materials from the Columbia-Mt. Pleasant, Tenn., area. Flotation studies were then extended to include selected low-grade washer products and raw ores from the district, and to determine the response of these materials to the simple flotation conditions used in beneficiating the waste-pond material.

In summary, the results of these experiments successfully demonstrated the ability to treat the early waste-pond materials, the low-grade washer products, and certain types of raw Tennessee phosphate ore to yield substantially improved products. An anionic flotation technique, which is effective in the presence of slimes, should simplify the beneficiation of the Tennessee phosphatic materials.

The investigations suggest a high potential for recovering a large percentage of these waste materials as high-grade phosphate concentrates ranging from 60 to 82 percent phosphate.

Reclamation, Stabilization, and Disposal

Both mine and mill wastes that have no practical value are normally deposited at sites near the mining or milling areas in order to reduce disposal costs to a minimum. In very remote areas, wastes of this type are often deposited at the respective disposal sites with little or no effort toward containment or other environmental control measures. However, the tailings of some beneficiation operations are such that complete and effective stabilization is necessary to prevent natural water and air erosion from conveying the wastes into adjacent areas. The tailings residues generated at uranium leaching plants are of the type that should be kept within the disposal site boundaries because of their potential radiation hazard. Such hazards might also be reduced by modifying the processing plant systems to lower the amount of radioactive materials in the waste discharge.

Chemical Stabilization of Uranium Leach Plant Residues

In 1969, Havens and Dean (34) reported on the chemical stabilization of radioactive tailings generated at the Tuba City, Ariz., uranium leaching plant of the El Paso Natural Gas Co. El Paso Natural Gas requested technical assistance from the Bureau of Mines at the suggestion of the Arizona State Atomic Energy Commission, following a survey of the site by the South-West Radiological Health Laboratory of the Public Health Service, U.S. Department of Health, Education, and Welfare.

The site is located approximately 6 miles east of Tuba City, on the Navajo Indian Reservation in Coconino County, Ariz. The radiological survey had reported disturbing radiation levels in the tailings disposal piles and nearby areas where windborne tailings dust had been deposited.

An average gamma radiation of 0.7 milliroentgen per hour was measured at a distance of 3 feet above the entire tailings area. According to the survey, this radiation level translates to an average annual dosage of 6 rem (roentgen equivalent man) for continuous exposure or 12 times the acceptable radiation level of 0.5 rem/yr for continuous whole-body exposure. Radiation measurements of the windborne deposits within 1,000 feet of the disposal area property fence and 3 feet above the surface were found to be equivalent to a continuous-exposure annual dosage equal to or greater than the 0.5-rem/yr level cited in the radiation protection guide (RPG)[4]. The mill area measurement of gamma radiation levels averaged nearly two times that of the RPG.

The concentration of radon-222 in the atmosphere adjacent to the tailings deposits was far below the radiation concentration guide (RCG) level of 3 picocuries per liter (pCi/l). The radon concentration directly over the pile was only slightly below the PCG. Sampling downwind airborne particulate concentration near the tailings indicated insignificant levels of radium-226, but levels of three times the RCG (200×10^{-5} pCi/l) for insoluble radium were found at distances up to 200 feet east of the mill property.

However, at the time of the survey, there was no evidence of surface or ground water contamination caused by leaching or radioactive materials from the tailings.

The objective of the Bureau of Mines stabilization studies was to determine the most practical and effective method for stabilizing the uranium leach plant tailings that had accumulated at a site 5,000 feet above sea level, where the temperature ranged from 110° F in summertime to -15° F in the winter and the normal annual precipitation was about 6.5 inches.

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[4] The radiation protection guide and the radiation concentration guide are used to calculate radiation concentrations from various radioactive materials and equivalent radiation various units. The guides are found in the Radiological Health Handbook (revised edition), published by the Consumer Protection and Environmental Health Service, U.S. Department of Health, Education, and Welfare. It is available from National Technical Information Service, Springfield, Va., PB 230 846.

After concluding numerous laboratory-scale stabilization tests on materials and methods, with suitable evaluation techniques and appropriate controls, chemical stabilization was determined to be preferable to attempting vegetation cover because of the composition of the surface materials at the site and the relatively harsh climate for vegetative growth.

The two chemicals selected for full-scale stabilization were DCA-70, a Union Carbide Co. elastomeric polymer; and Lorlig A, an American Can Co. calcium magnesium lignosulfonate. The former was selected because of its good penetration (up to 2 inches) into the sandy-textured dyke materials, and because of its transformation into a very hard water-insoluble crusty surface soon after application. The latter material was selected because of the extremely hard surface crust it produced on both the acid and carbonate tailings. It was also the only one of all chemicals tested to produce hard crusts having durable wind resistance when applied to the carbonate tailings.

The procedures for stabilizing the ponds, dykes, and nearby adjacent areas included preliminary mixing of chemicals with water in a large vat, and the application of the pumped solutions through an automatic, traveling sprinkler. The chemical applications were varied from 0.045 to 0.068 gallon of liquid DCA-70 per square yard for the dyke areas or an average of 0.53 gallons per square yard at a cost of 9.03 cents per square yard. The application of Norlig A (dry powder) to stabilize tailings areas was varied from 0.1 to 1.2 pounds per square yard of surface. The average application was 0.93 pound per square yard at a cost of 4.5 cents per square yard.

The direct cost for complete stabilization of the 34.5 acre site was about $335 per acre. All treated surfaces appeared to have been adequately stabilized for a reasonably long time at a reasonably low cost.

Reduction of Radioactive Materials in Uranium Mill Tailings

One way to reduce the problem of stabilizing uranium mill tailings is to reduce the amount of radioactive materials carried in liquid form with the mill tailings into the disposal areas. Tame, Valdez, and Rosenbaum (86) began a study of this problem in 1961 by investigating the flowsheets employed at the major uranium mills in search of procedural changes that would reduce the amount of radioactive materials carried with the tailings into the disposal areas. One of their reports pertains to the Kerr-McGee Oil Industries, Inc., process at Shiprock, N. Mex.

The radioactivity of the uranium ores supplied to the mill stems from a secular phenomenon in which the natural isotope, uranium-238, decays very slowly to lead-206 by the release of alpha and beta particle emissions. The uranium is normally in secular equilibrium with its daughter products but this equilibrium is upset when the uranium is leached from the ore.

The radioactive materials normally found in liquid processing wastes are shown in table 1, which includes the half-lives, modes of emission, and permissible concentrations of the various constituents.

TABLE 1. - Permissible concentrations of radionuclides in water discharged to unrestricted areas (94)

Isotope	Half-life	Radiation	Permissible, μCi/ml	Concentration, dpm/l
Uranium, natural.......	-	Alpha.....	2×10^{-5}	4.4×10^{4}
Thorium-234...........	24.1 days	Beta......	2×10^{-5}	4.4×10^{4}
Thorium-230...........	8 10^{4} years	Alpha.....	2×10^{-6}	4.4×10^{3}
Radium-226............	1,622 years	...do.....	1×10^{-8}	22.2
Polonium-210..........	138.2 days	...do.....	7×10^{-7}	1.6×10^{3}

Concentrations are given in units of microcuries per milliliter (μCi/ml) and in disintegrations per minute per liter (dpm/l). To convert dpm/l to μCi/ml divide by 2.2×10^{9} (91).

The Bureau of Mines study, which was prompted by the results of an Atomic Energy Commission-sponsored survey of the U.S. uranium mills, showed that the liquid effluents of all mills exceeded the permissible concentrations of radium-226, and the levels of soluble uranium and thorium-230 were excessive in some individual sample materials.

The proposed Bureau of Mines method of reducing surface and subsurface water pollution by radioactive mill wastes was to repeatedly recycle part of the process waste solution back to the washing circuits to supplant the normal supply of fresh water (91).

In the Shiprock uranium-vanadium extraction process, crushed ore was first leached with sulfuric acid in two stages. This was followed by organophosphate solvent extraction in which uranium and vanadium were recovered from pregnant leach liquor in sequential stages. Wastes from the acid-leaching stages were comprised of slimes and sands ultimately discharged from the four thickeners and four spiral classifiers, respectively. Some solution still associated with the sands and slimes was also discharged into the waste disposal area. The ultimate reduced pregnant solution obtained by leaching was supplied to the solvent extraction section of the plant, which comprised four stages of uranium extraction and three stages of uranium stripping, followed by five stages of vanadium extraction and four stages of vanadium stripping. Vanadium and uranium products were precipitated from their respective concentrated stripping solutions. The uranium-barren solution (raffinate) from the first four stages of uranium solvent extraction was delivered to the first of the five-stage vanadium solvent extraction. The uranium-vanadium barren solution (raffinate) discharged from the first five stages of vanadium solvent extraction still contained some radioactive constituents, but the normal practice was to discard this solution along with the tailings.

The study was designed to determined the effects of recycling some of the raffinate back into the washing circuits in place of equivalent fresh water makeup. The laboratory operations duplicated the Shiprock process except for the laboratory recycling of raffinate back into the system through consecutive cycles.

The experiments demonstrated that reuse of solvent extraction raffinate in the laboratory duplicate of the Shiprock process had no adverse effect in the liquid-solid separations, washing of leached residues, or solvent extraction of uranium. The concentration of radium-226 in recycled solutions did not increase with continued recycling. The benefits of recycling the raffinate to washing circuits included (1) reduction in the amount of radium in discharged waste effluent by about 75 percent, (2) reduction of acid requirements for leaching, and (3) reduced fresh water requirements.

Chemical and Vegetative Stabilization of Copper Mill Tailings

Wastes from copper beneficiation mills are accumulating by the billions of tons annually. These wastes, like other low-value materials, have so little potential value that stabilization of disposal areas appears to be the only practical means for minimizing air, land, and water pollution caused by these wastes. A 1969 report by Dean, Havens, and Harper (23) describes an investigation of two methods for stabilizing the tailings at McGill, Nev., produced during the flotation of porphyry copper ore at the Kennecott Copper Corp. mill. Results of previous stabilization studies by Kennecott Copper Corp. indicated earlier vegetative stabilization failures were the result of windblown sand particles abrading and burying the plants emerging from previously seeded areas. The Bureau of Mines, with the assistance of Kennecott Copper Corp., demonstrated that a commercially available resinous adhesive chemical would effectively bind the fine tailings particles to form a stable surface crust without interfering with the plant growth processes. The demonstration was a combination chemical-vegetation stabilization scheme at a Nevada test area 6,340 feet above sea level where the normal growing season is about 74 days. In preliminary tests, the tailings were found to contain about 12 percent iron and 62 percent SiO_2 with much lesser amounts of aluminum, magnesium, potassium, sodium, and calcium oxides, and about 2.4 percent sulfur. The moisture averaged about 13 percent. It was also found that the surface area at the site was sufficiently light in color so that it reflected solar radiation to the under surfaces of the plants and caused some physiological stresses. The talings also contained some salts and phytotoxicants that normally inhibit plant growth. The salt content (0.38 percent) is normally considered to be moderately salty by agronomists.

The objective of the stabilization investigation was to produce a first vegetative cover that would renew itself and thereby provide suitable long-term stabilization of the areas by the eventual encroachment of indigenous vegetation species.

After a thorough study and evaluation of the soil, the environment, and potential starting species, 11 plant types were selected for seeding: winter wheat, Ladak and Ranger alfalfa, yellow sweet clover, Siberian pea tree, Russian wild rye, and western, crested, intermediate, tall, and pubescent wheat grass.

Tests with a wide variety of chemical stabilizers demonstrated the superior quality of the resinous adhesive reagent Coherex as a practical cement for all types of tailings surface conditions at the site, in addition to having minimum interference with plant growth.

Preliminary conditioning of the tailings for plant growth was accomplished by adding nitrogen and phosphorus in the form of prilled urea and calcium treble superphosphate at a rate equal to 45 pounds per acre of nitrogen and P_2O_5.

One month after the addition of Coherex, seeds, and fertilizer, plant counts showed that 52, 24, and 12 percent of the winter wheat, legumes, and grasses, respectively, had germinated. An early freeze prevented further studies during the remainder of that season. However, in a survey of the area 1 year later, it was found that the original objective had been achieved, and a wild plant cover had developed and was flourishing.

Subsequent evaluation studies, based on a Coherex dilution to four parts by volume water, indicated a stabilization cost of $135.50 per acre, which was considered to be acceptable.

PROCESSING BULK RAW MATERIALS

In 1974, the raw materials beneficiation industries received bulk supplies of crude petroleum, coal, gas, iron ore, bauxite, sand and gravel, stone, and other natural domestic resource materials valued at $55 billion (97). The products of these industries, plus $20 billion worth of imported petroleum, iron ore, bauxite, etc., and $4 billion worth of recycled domestic materials such as glass, scrap iron, aluminum, lead, zinc, and other secondary materials were supplied to the bulk raw materials processing industries. These processing industries include melting, refining, and energy generation and transmission, which together produce energy and processed materials of mineral origin valued in 1974 at $210 billion. The products of the bulk raw materials processing industries include commodities such as fuels, electricity steel, aluminum, chemicals, plastics, fertilizers, and many others normally supplied as raw materials, to the manufacturing industries. Typical wastes include metal smelter and refiner dusts, smoke, fume, drosses and slags, electric utility fly ash, spent liquors, and solutions from metal-treating operations, and many other solids, solutions, and gaseous wastes that differ widely in physical character, contained values, and quantities generated.

A significant part of these wastes is currently recycled to both the primary and secondary materials industries, together with substantial amounts of postconsumer goods such as glass, paper, scrap metals, plastics, and others However, large amounts of the wastes such as slag, fly ash, and many processing solutions contain so few recoverable values that they must either be disposed of as economically as possible by nonpolluting techniques, or new methods must be found for extracting some of the values to help pay for rising disposal costs. The following discussions pertain to selected investigations on these materials and the potential for improved recovery, utilization, and disposal techniques.

Recovery

Values From Zinc Smelter Residues

Horizontal zinc distillation retorts have been employed for many years to supply a substantial part of the annual U.S. demand for primary zinc. Retorting is a batch process for reducing zinc minerals to the metallic state and distilling it away from its host mineral constituents, leaving a residue that generally contains significant amounts of recoverable copper, silver, iron, and residual zinc. Since these metals are all associated with iron, it is possible to separate them from nonmagnetic constituents in the residue by magnetic concentration techniques. Sizable tonnages of these retort residues have accumulated during the past 40 to 50 years.

In 1973, Powell and Higley (65) reported their extensive investigation of the retort residues and improved methods for recovering the contained values.

Typical residues were found to contain unburned carbon, shot and stringers of metallic iron, copper inclusions in the iron as both metal and sulfides, silver in solid solution in the copper, and glassy slag. Common practice at distillation smelters was to crush the cooled residues and magnetically separate the magnetic and nonmagnetic constituents. The magnetic fraction was an upgraded copper-silver material normally suitable for shipment to a copper smelter for silver and copper recovery. If magnetic materials were too low in grade for processing, they were stockpiled. In 1969, the total estimated amount of magnetic residues generated was 200,000 tons. Based on 1972 prices, the total value of zinc, copper, silver, and iron in these residues was estimated to be about $125 per ton. At 1972 prices, the magnetic fraction of the zinc-retort residues generated in 1969 would contain nearly $25,000,000 worth of zinc, copper, silver, and iron.

In preliminary experiments designed to evaluate promising areas for subsequent in-depth research, pyrometallurgical processing methods were found to show greater promise than conceivable chemical leaching or physical separation and concentration of metallized constituents. In a series of pyrometallurgical tests, it was found that either reduction-smelting or matte-smelting methods would recover essentially all of the zinc, copper, silver, and iron contained in the magnetic fraction of retort residues. Zinc was vaporized and recovered as zinc oxide by either method.

Reduction smelting without addition of sulfur or flux produced a small amount of rich matte and high-copper iron alloy. The matte contained nearly all of the silver, some of the copper, and a small amount of the iron; the remainder of these metals were in a high-copper-iron alloy. These products of reduction-smelting materials were thought to be suitable for use in commercial copper-precipitation plants as a substitute for scrap iron. In such application, the copper and silver might be recovered from both the matte and the high-copper-iron alloy.

Matte smelting with pyrite produced a matte containing nearly all of the silver, 80 percent of the copper, and 37 percent of the iron. The remaining copper was recovered with iron in a coproduct alloy.

The copper could be recovered from the copper-iron alloy with molten lead or it could also be removed with sodium sulfate additions. But lowering the copper from 6.28 to 0.05 percent resulted in a 25-percent loss of iron to the slag.

It was concluded that esentially all of the metal values in the magnetic fraction of the residues could be recovered by appropriate processing. The total value of recovered constituents suggested possibilities for favorable economics.

Values From Brass Smelter Dusts

The producers of brass and bronze alloys also collect considerable amounts of dust in various types of collecting systems, frequently of the baghouse type. Although the composition of brass-smelting dusts vary considerably at different plants, most of them contain substantial amounts of zinc and lesser amounts of lead, copper, tin, and iron. As yet, there have been virtually no attempts to recover the metal constituents, largely because of unfavorable economics. However, it has become more important to reconsider all possibilities for processing these dusts in recent years because of the more stringent disposal regulation imposed to prevent air, land, and water pollution from this source. The amount of brass smelter flue dust generated in the United States now averages about 2 tons per day per smelter or a total of approximately 1,560 tons per day. A 1974 report by Fukubayashi and Higley (28) discusses an investigation of potential processes for the economic extraction of zinc and lead from these materials.

The sample materials obtained were typically too light and fluffy for shipment to a prospective user. Dusts from the different sample materials contained 47 to 71 percent zinc, 2.6 to 7.0 percent lead, 0.54 to 0.84 percent copper, less than 0.1 to 1.2 percent tin, less than 0.1 to 0.45 percent iron, and 0.34 to 2.49 percent sodium. The sulfur content ranged from 2 to 6 percent, chlorine from 0.4 to 2 percent, and carbon from 0.7 to 2.7 percent.

Various mixes of dusts and charcoal reductant were furnaced at 1,050° C to reduce and vaporize the metals. Different mixtures were furnaced in unpacked and pelletized form. Studies were made of zinc volatilization rates and the effects of pelletizing and the partitioning of zinc and lead in the volatilized products. The effects of sodium and chlorine on the vaporization process were also investigated. Experimental results showed that a significant part of the lead and essentially all of the zinc can be reduced and vaporized from brass smelter flue dusts in an oxygen-free reduction at 1,050° C. The wood charcoal used as a reductant (up to 10 percent) contained impurities such as CaO and FeO, which catalyze the reduction of ZnO. The reduction-volatilization process was enhanced by pelletizing the mixed dusts and reductant. Optimum pellet size appeared to be 0.6 inch in diameter for minimum time and charcoal requirements. Additions of 4 percent $NaOH$ increased zinc vaporization markedly, but equal amounts of NaCl increased the vaporization of both zinc and lead. Nearly 95 percent of the zinc was volatilized in 4 hours, but no more than 40 percent of the lead could be removed regardless of time.

Values From Steelmaking Wastes

Certain operations in modern steelmaking processes involve very energetic, sometimes violent reactions between injected air or oxygen and the constituents in molten iron or steel charges. These reactions convert a small fraction of the molten metal constituents to a fine dust or fume, most of which is collected in equipment designed for that purpose. At the present level of steel production in the United States, the estimated production of steelmaking dust exceeds 2 million tons per year. In addition to considerable quantities of iron, the dusts also contain substantial quantities of lead, zinc, and manganese. Significant amounts of iron are also contained in the mill scale during the forming and shaping of hot steel materials.

Recovery of lead and zinc from the steelmaking dusts and mill scale, and the production of prereduced pellets for recycling to upstream iron and steelmaking operations, was the subject of a 1974 report by Dressel, Barnard, and Fine (24). After several preliminary investigations, they ruled out acid-leaching processes as being too impractical, and concluded that the most practical approach appeared to include the following steps: (1) pelletize untreated dusts, (2) remove lead and zinc by roasting under controlled conditions, and (3) reduce the roasted pellets to produce an upgraded iron product for recycling. Because of the low levels of copper in the dusts (0.30 percent), copper contamination of the products was not considered a significant problem. Original plans called for the addition of coke breeze to the dust as a reductant and the addition of mill scale because of its relatively high iron content. Table 2 shows the percentages of various elements in the materials investigated.

TABLE 2. - Composition of test materials, percent (24)

Element	EF	BOF	BF	MS	CB
Fe	33.2	55.7	29.0	73.0	1.5
Pb	2.6	.4	<.05	-	<.05
Zn	24.8	6.3	.15	-	.02
Cu	.27	.03	.02	-	.01
Mn	3.3	.54	.34	-	<.10
Ca	.92	3.8	6.7	-	<.05
Si	1.6	2.2	5.4	-	2.0
C	.30	.44	25.4	1.3	80.7
S	.48	.11	.54	-	.90

NOTE.--EF--electric furnace dust.
BOF--basic oxygen furnace dust.
BF--blast furnace dust.
MS--mill scale (waste).
CB--coke breeze.

The research procedures included a variety of pelletizing and roasting techniques for zinc and lead removal, followed by a series of iron metallization tests to determine the best types of equipment and optimum temperature and carbon requirements.

It was found that steelmaking dusts and mill scale can be pelletized and thermally treated to remove lead and zinc, and then reduced to convert the contained iron oxide pellets into metallized iron pellets. More than 99 percent of the zinc and 85 percent of the lead was volatilized from the dust by roasting in a carbon monoxide atmosphere at 1,000° C. The volatilized lead and zinc was conveniently collected for further processing.

Reduction and volatilization of over 95 percent of the zinc and most of the lead was accomplished in a 1,100° C roast of self-reducing mixtures of steel furnace dusts and high-carbon steelmaking waste (mill scale) in an inert atmosphere. Over 96 percent of the iron in the resulting pellets was metallized and well suited for recycling. A study of potential commercial application of the process suggested a central processing plant where large amounts of dust (200,000 to 400,000 tons annually) would be available within a 50-mile radius. Operating on a lesser scale appeared to be impractical.

Copper From Converter Slags

For many years, most domestic copper has been produced from sulfide ores in a series of furnace reduction, conversion, and refining steps. The first step is the reduction of copper mill concentrates in a reverberatory furnace to form a copper matte (copper-iron-sulfur compounds). Semirefined copper (blister) is produced from the matte in a converter furnace in which the unwanted sulfur and iron are separated by oxidation. Blister copper, sulfur dioxide, converter flue dust, and converter slag are formed in the operation. The converter slag normally has too much copper (normally 1 to 5 percent) to be discarded, so it is returned to the reverberatory furnace for repeated processing with additional concentrates. Unfortuantely, this practice complicates reverberatory furnace operations and contributes to the loss of some copper in discarded reverberatory furnace slag. Edlund and Hussey (27) investigated an alternative procedure in which they proposed re-treating converter slags by flotation to recover copper concentrates rather than the conventional, relatively low-grade converter slag, for recycling to the reverberatory furnace. The copper in the concentrates was fine particles of copper sulfide. If successful, the practice would simplify reverberatory furnace operation, increase its capacity, and reduce the copper losses.

Batch flotation tests were designed to determine the relative merits of pretreating the various test slags by slow cooling or water quenching to form copper-containing structures that would be easily liberated and most amenable to flotation concentration. The three selected test slags contained 1.6, 5.0, and 6.6 percent copper.

By grinding and floating the slow-cooled slags, more than 90 percent of the copper was recovered in prills and in these rougher concentrates. The resulting tailings contained 0.2 to 0.3 percent copper. When floating water-quenched slags, copper recovery was lower, and the tailings contained 0.5 to 0.6 percent copper. It was also found that slow-cooled slags were less difficult to grind than the water-quenched slags. However, cost studies indicated that the high cost of slow cooling the slag tends to cancel the advantages of higher copper recovery. Since water-quenching the slag precluded the need of grinding prior to flotation, this practice would be the most practical. Cost studies based on a plant capacity of 1,000 tons of slag per day indicated a processing cost of \$1.86 per ton of quenched slag and \$2.37 per ton for the slow-cooled slag.

Materials Utilization

Utilizing Phosphorous Furnace Slag for Making Ceramic Tile

A 1974 report by Valdez, Dean, and Warner (99) describes research investigations on the utilization of phosphorous furnace slag in ceramic wall and floor tile. The incentive for the investigation stemmed largely from the annual 4 million tons of phosphate rock mined in the State of Idaho and processed in electric furnaces, and other equipment to produce phosphoric acid, elemental phosphorus, and various types of fertilizers. Reports claimed that more than half of the mined phosphate rock was consumed in the production of elemental phosphorus at plants of FMC Corp., Pocatello, Idaho, and Monsanto Co., Soda Springs, Idaho. A flux of silica rock was added to the phosphate rock to form a charge mix for the electric furnaces, which annually generate about 2 million tons of electric phosphorous furnace slag.

The slag was found to have a composition corresponding to a pseudowollastonite known as the alpha form of natural wollastonite, a mineral mined in large tonnages to supply the ceramic tile industry. Since there had been no previous commercial use for the phosphorous furnace slag, an investigation was initiated to determine the possibility of using the slag as a substitute wollastonite for making wall and floor tile (99). The FMC Corp. supplied furnace slag for the investigations from its Pocatello plant. The color of the slag ranged from near black to light gray. Microscopic examination revealed a predominantly pseudowollastonite composition ($CaSiO_3$) with minor contamination by other silicates. Also present were scattered metallic grains of chromium, vanadium, iron, nickel-copper, and phosphorous alloys. Chemical analysis indicated about 45 percent SiO_2, 42 percent CaO, 6 percent Al_2O_3, 3 percent iron, and less than 1 percent each of K_2O, Na_2O, MnO, S, and P.

Typical wall tile mixes are comprised of 80 to 85 weight-percent of wollastonite and 10 to 20 weight-percent ball clay and flux. Floor tile mixes generally contain about 55 percent feldspar, 10 percent flint, and 35 percent clay. Commercial floor and wall tile must meet industry specifications for shrinkage, water absorption, and physical strength.

Experimental procedures were planned to determine the effect of particle size, firing time and temperature, impurities, and different mix compositions. Also included were measurements of physical properties. A cost evaluation of the process was made to determine the economic potential for commercial application of the process.

The research demonstrated that electric phosphorous slag could be used to produce high-quality tile products. When properly ground and treated magnetically to remove iron constituents (magnetite), the slag comprised a raw material suitable for forming, dry pressing, sintering, and glazing to yield quality floor and wall tile meeting ASTM specifications C34-62 for structural clay, load-bearing tile. A commercial-grade tile could also be made by adding borax to the slag, followed by dry pressing and firing. The estimated production cost of $0.43 per square foot of wall tile compared favorably with the cost of commercially produced wall tile. The use of furnace slag for tile production could preclude the need to mine thousands of tons of natural wollastonite presently used for that purpose.

Sulfur in Special Concretes

Recent regulations setting low permissible levels of sulfur released to the atmosphere have been followed by a flurry of new efforts to collect sulfur and sulfur compounds from processing and manufacturing plants effluents. In some situations, it has been profitable to convert the collected sulfur constituents to sulfuric acid. However, the total quantity of the sulfur-containing waste materials likely to be collected during the next decade will more than supply the normal demand. Accordingly, considerable research was aimed at finding methods to convert these collected waste materials to elemental sulfur, which could then be available in large quantities for other purposes. This situation prompted additional investigations aimed at finding new uses for the excess sulfur. One such study involved the use of elemental sulfur in structural materials. A project by Crow and Bates (28) was conducted under a National Aeronautics and Space Administration contract to the Bureau of Mines for the primary purpose of studying various aspects of lunar sampling and mineral exploitation. The specific work of interest was to advance the ground-support materials and technology for both terrestrial and extraterrestrial mining. Sulfur was used in a wide variety of mixes and preparation to make strong and durable structures. These investigations are reported here only because of their potentially economic uses for large supplies of sulfur on earth that may greatly exceed normal demands. The investigations were designed to demonstrate the value of sulfur for bonding well-graded basalt aggregate mixes to form high-strength sulfur-basalt concretes.

The sulfur was found to be an excellent bonding agent, and the strength of thermoplastic sulfur aggregate mixtures was governed primarily by the grain-size distribution in the aggregate.

The mixtures were prepared in an electrically heated 3-cubic-foot mixer and the strengths of various concretes were determined by breaking test cylinders 6 by 12 inches. Average strengths ranged from 3,348 to 10,398 pounds per square inch. The highest compressive strength cylinder was measured at 10,717 pounds per square inch.

Experimental results indicate the following:

1. A double-graded aggregate was essential for obtaining maximum strength and facilitating mixing. The large particles gave strength and provided workability; the fine particles filled voids and provided maximum bonding surface.

2. The addition of 10 percent minus 200-mesh material added strength by holding the proper amount of sulfur in suspension.

3. The problem of shrinkage was serious but could be reduced by continuing to add mixed material into the shrink space as the mold cooled.

4. Proper control of aggregate particle size distribution in sulfur-basalt concretes produced compressive strengths greater than 10,000 pounds per square inch.

5. Because sulfur softens at relatively low temperatures and also has an appreciable vapor pressure, concretes with sulfur binders would not be suitable for long-term structures in hot, low-pressure environments.

Values From Alumina Industry Red Mud

In 1970, Fursman, Mauser, Butler, and Stickney (30) reported on an investigation of potential methods for recovering values from the mud waste generated by the alumina-producing industry. The investigation was aimed at reducing the tremendous annual tonnage of mud that must be stored on land areas or discharged into rivers. In 1967, more than 6.8 million tons of mud (dry basis) was generated by eight alumina plants located in central Arkansas, on the gulf coast, and along the lower Mississippi. Red mud is the residue left after contained alumina has been extracted from the aluminum ore, bauxite. The types of bauxite from which red mud originates include the Jamaican types from Jamaica, Haiti, and the Dominican Republic, and the Surinam-types from Surinam and Guyana. The bauxites are treated by the well-known Bayer process to obtain alumina (Al_2O_3) for the ceramic and aluminum-producing industries. The third type of bauxite comes from Arkansas and is normally processed with some red mud from a Bayer operation in a combination of steps, including sintering with lime and soda to produce water-soluble aluminates and then leaching with water to recover the aluminates. The residue of this process is known as brown mud. The types of bauxite processed, the amount of red mud generated, and the amounts of various constituents are shown in table 3.

The investigations included studies on Jamaican and Surinam red muds for the extraction of iron and alumina by electric arc furnacing and a lime-soda reduction sintering operation. The sinter was water leached to extract soluble sodium aluminate. An iron product was recovered from the leached residues by magnetic separation. The investigations included both bench- and pilot-scale studies on the sintering method. A pig iron product and a calcium aluminate slag were produced in the electric arc furnace, and a sodium carbonate solution was then used to leach alumina from the calcium aluminate slag.

Cursory investigations included the recovery of TiO_2 from Surinam red mud sinter-leach residues and combining brown mud and leached calcium aluminate slag to produce portland cement.

TABLE 3. - Estimated production of alumina-plant muds and contained minerals in 1967 (30)

Bauxite source	Mud type	Residues and mineral contents, short tons					
		Total mud[1]	Al_2O_3	Fe	TiO_2	Na_2O	CaO
Jamaican...	Red........	4,257,400	851,500	1,417,900	276,700	170,300	212,900
Surinam....	Red........	1,286,300	257,300	225,100	128,630	133,800	77,200
Arkansas...	Brown......	1,298,800	115,600	71,400	45,500	70,100	519,500
Total............		6,842,500	1,224,400	1,714,400	450,830	374,200	809,600

[1] Dry basis.

It was found that 85 to 90 percent of the alumina could be recovered in the sinter-leach solution. Since the ratio of silica-to-alumina was too high to produce cell-grade alumina, a desilication treatment would be required. In a commercial operation, this solution could be recycled to a process such as the Bayer liquor-recycle system.

From 75 to 90 percent of the iron was recovered from Jamaican red mud by magnetic separation to yield a magnetic product containing up to 55 percent iron. This product would be suitable for making foundry pig iron, or it could be refined to steel by first oxidizing and removing the carbon and phorphorus. It was found that the sintering process would dispose of about 70 percent of the red mud.

A 96-percent titania product was extracted with sulfuric acid from the nonmagnetic fraction of the Surinam red mud, which originally assayed up to 20 percent titania. Titania recovery of 87 percent was achieved.

It was also found that an electric-arc-furnace process could be employed to recover an iron product and leachable calcium aluminate slag from the Jamaican and Surinam red muds. Up to 98 percent of the iron was recovered in a regulus and up to 84 percent of this alumina was recovered by leaching the slags with sodium carbonate solution. The leached calcium aluminate slags and brown mud were found to be suitable essential ingredients in the production of portlant cement.

Fly Ash for Water Clarification

Coal was burned to supply 18 percent of the total U.S. energy demand in 1974. This amounted to 13.2 quadrillion Btu (97). Burning that amount of coal generated an enormous amount of fly ash, which was disposed of at a substantial annual cost.

A comprehensive 1972 report on the generation and consumption of fly ash was made by Covey and Faber (20) who predicted a 1980 U.S. coal consumption of nearly 490 million tons and a corresponding fly ash production of 35 million tons. However, 1974 data (69) compiled by the National Ash

Association and Edison Electric Institute show the total ash collected was 59.5 million tons, which exceeded the amount for 1973 by 10.2 million tons. Of the 1974 total, 40.4 million tons was fly ash, and 14.3 million tons was bottom ash, with a balance of boiler slag. The ash utilized in 1974 was 8.7 million tons, 0.7 million ton greater than the amount used in 1973. Half of the boiler slag, 20.3 percent of the bottom ash, and 8.4 percent of the fly ash was utilized in 1974.

Numerous schemes have been proposed for utilizing more of the fly ash in order to reduce disposal costs. One potential use, investigated by Ballance, Capp, and Burchinal (5), was that of fly ash as a coagulant aid for the clarification of turbid water. Coagulation of suspended matter with alum is currently common practice for clarifying turbid wastes for domestic and industrial use.

One of the first coagulant aids (sodium silicate) was studied in 1935-36 (8). Since then, other materials have been investigated including bentonite clay (60), cement dust (29), and polyelectrolytes (52, 68).

Commercial use of fly ash has generally been restricted to applications such as in lightweight aggregate, for soil stabilization, and as a concrete additive. No previous work had been reported on its use as a coagulant for treating turbid water.

Balance, Capp, and Burchinal (5) followed standard laboratory procedures to determine the performance of fly ash when applied for clarifying synthetically turbid stock water. The supply of stock water was prepared by filtering a suspension of kaolinic clay through coarse filter paper in a Buchner funnel. In each jar settling test, turbid stock was diluted with distilled water to provide test samples containing 100 ppm of materials in working suspension. The settling rates of the suspended materials were measured for a wide variety of conditions. These tests were followed by floc-formation tests, which included measurements of settling rates and sludge densities corresponding to various additions of fly ash and other materials. Results of these tests were compared with control tests in which only alum was used.

The results indicated that fly ash from several different sources, when applied in proper proportions, assisted the chemical coagulation of constituents in turbid water and in the settlement of the induced floc. Observations of specific effects included a reduction of time for visible floc to form and an increase in the mean rate of settling flocculated particles. The fly ash was observed to promote a more uniform floc size, which appeared to be one reason for the settlement of denser sludges

The final turbidity of the water was not effected by the fly ash particle size or its carbon content.

Development of Processes and Techniques

Drying and Carbonization of Wood Wastes

Large tonnages of U.S. lumber mill wastes such as chips, shavings, mill pieces, and sawdust are destroyed each year by burning. Although energy is recovered from some of the unburned wastes, substantial amounts are burned without any benefit other than disposal. It is very probable, however, that very large amounts of these wastes could be used in metallurgical, chemical, and other processes if they could first be economically dried and carbonized. A study of techniques for doing this was conducted by Boley and Landers who reported on their investigations in 1969 (9).

Their objective was to evaluate the application of entrainment drying and carbonization techniques that had been demonstrated successfully for drying and carbonizing coal. The yields of carbonization at corresponding selected experimental conditions were to be determined. A secondary objective was to determine the assay carbonization characteristics of these wastes and how well the resulting charcoals could be briquetted.

The sample materials for the tests included both hardwood and softwood wastes supplied by the Silver Corp., Silver Engineering Works Division, Denver, Colo.

The softwood waste was comprised of redwood planer shavings, green planer shavings, and green resaw sawdust. The hardwood waste was oak chips, some as large as 4 by 2 by 2-1/2 inches. Test materials were prepared by separately reducing 3,000 pounds of redwood and 2,000 pounds of oak wastes in a hammer mill to 1/4 inch or less. The heating value of these prepared wastes was found to be 9,200 Btu/lb for the softwood and 8,550 Btu/lb for the hardwood.

Preliminary carbonization assay tests were made to determine the maximum obtainable carbonization yield that could be expected by any processing technique.

Entrainment drying was performed by entraining the wastes in a moving stream of hot gases, mostly nitrogen and water vapor, until they were dry, at which time they were recovered from the gas stream in a cyclone-type separator. The hot gases were generated in an external combustion chamber using natural gas fuel. The risk of explosions in the drying system was precluded by burning with a deficient supply of oxygen.

Two drying units were used--one 6 inches in diameter and 5 feet, 4 inches long, and the other 12 inches in diameter and 8 feet long.

Entrainment carbonization was performed in an 8-inch-diameter by 16-foot-long carbonizer. The waste particles were carried upward in the column in a turbulent gas stream of combustion gases and volatile carbonization products. The total supply of waste was first dried to about 2 percent moisture in the entrainment dryer with heat supplied from an external heater.

Four experimental tests were made in the entrainment carbonizer with external heat supplied. The feed rates of dried wastes ranged from 125 to 152 pounds per hour. In three tests, makeup heat (for system losses) was supplied from an external source. The charcoal yield for these tests ranged from 22.5 to 27.4 percent of the dried waste. One test in which heat for carbonization and system losses was supplied internally produced only 16 percent charcoal. Volatile constituents (excluding bound moisture) in the four charcoal products ranged from 24 to 30 percent.

Satisfactory briquets were made from the oak charcoal but a large amount of starch binder was needed. The redwood charcoal could not be briquetted.

Dewatering Jamaican Red Mud

The red mud waste generated in the Bayer process for recovering alumina from bauxite contains substantial amounts of process solution which add to the problems of storage or other disposal. The red muds originating from Jamaican bauxites are particularly troublesome because they are not readily amenable to filtration and thus difficult to dewater before they are discharged to disposal sites. These muds are characterized by a high iron content and very small average particle size (50 percent of the particles are smaller than 6 micrometers). The solids content of the average Jamaican red mud is about 20 percent. In commercial practice, the mud is discharged into large artificial ponds and, in some cases, it is discharged into navigable waters under the supervision of the U.S. Army Corps of Engineers.

Good and Fursman (31) reported on centrifuged dewatering Jamaican red mud in 1968. The reasons for dewatering included the following:

1. Reducing the bulk of materials discharged from the process plants, thereby increasing waste-storage pond capacity. Furthermore, dewatering would reduce transport charged for operations that are required to move the mud to remote disposal sites.

2. The clarified liquid, recovered by centrifugation, contains significant quantities of valuable alumina and soda in solution that could be recovered by recycling the recovered solution to the Bayer process in lieu of makeup water.

In batch tests with a small laboratory centrifuge, it was found that the maximum suspended-solids content of centrifuge residues was 47.5 percent with a 60-second residence in a force of 1,625 gravity. By comparison, normal gravity settling for 10 days produced residues containing a maximum of 30 percent solids.

The experiments were then extended to include continuous centrifugation. The centrifuge was a standard counterflow type with a 6-inch-diameter chamber rotating about a horizontal axis and discharging clarified solution at one end and dewatered solids at the other. The test-condition variables included red mud feed rates, positioning of the feed stream, centrifuge pool depth, and centrifugal force. In some experiments, a flocculant was added to enhance the separation. Ultimately, the laboratory test data were extrapolated to a continuous 24- by 60-inch solid bowl centrifuge. The estimated capacity was 6,600 to 7,200 gallons per hour of red mud slurry which would contain about 7 tons per hour of solids on a dry basis. Cost analysis of an industrial application was based on 22 centrifuges processing 16,000 tons per day of Jamaican red mud slurry.

The results indicated that centrifuge dewatering on a commercial scale was feasible and significantly more effective than gravity dewatering. The estimated centrifugation dewatering cost for increasing solids from 20 to 40 percent was 9.4 cents per ton of slurry or $1,504 per day. Clarified solutions containing 130 tons of sodium carbonate and 41 tons of alumina trihydrate would be recovered daily. On the basis of September 1967 prices, recovered compounds would be worth 42.5 cents per ton of slurry or $6,800 per day.

Alloys From Stainless Steel Flue Dusts

Several types of waste are generated in substantial quantities during the production of stainless steel. Millions of dollars worth of scarce metals are lost annually in these wastes. A 1975 report by Powell, Dressel, and Crosby (64) cited annual estimated losses of chromium, nickel, and molybdenum amounting to 5 million, 1 million, and 90,000 pounds, respectively, in stainless steel furnace dusts. It was also estimated that the loss of these metals in other wastes such as grinding swarf and mill scale may be twice the amount of furnace dust losses.

The report describes laboratory-scale research on potential processes for converting wastes of this type to secondary alloys suitable for recycling back to the primary furnaces. Byproducts of zinc and lead, if present in the waste in sufficient amounts, would be recovered as oxide fume.

The types, quantities, and compositions of these wastes, generated during the production of stainless steel alloys, depends on the type of alloy produced, the alloying method employed, and the degree and manner of metal finishing. For example, four wastes are generated at a single plant operating electric arc furnaces in conjunction with the argon-oxygen procedure for making finished stainless steel products such as structural shapes, plates, rods, and bars. The four wastes include the electric arc furnace dust, the argon-oxygen furnace dust, grinding swarf, and mill scale. Other wastes of interest in the investigation included those generated at plants that produce ferroalloys other than stainless steel. Dusts collected at these plants are a mixture of various furnace dusts that contain high-value ferroalloy elements in substantial amounts. An estimated 40 pounds of stainless steelmaking dusts is generated for each ton of stainless steel produced, or more than 24,000 tons of dust per year. The amount of mill scale and grinding swarf generated at a typical plant was estimated at 70 percent of the total waste produced.

The major elements in a composite of five different electric arc furnace dusts, with one argon-oxygen furnace dust, assayed slightly more than 23 percent iron, about 8 percent chromium, 2 percent nickel, 5 percent manganese, 3 percent zinc, about 2 percent lead, and slightly over 2 percent carbon. The major elements in another composite sample of electric arc and argon-oxygen dusts, with mill scale and grinding swarf, assayed 46.5 percent iron, 10.5 percent chromium, and 3 percent nickel. It also contained 1 and 8 percent zinc and lead, respectively. Manganese and molybdenum were present in amounts of 1.4 and 0.4 percent, respectively.

Laboratory investigations included preliminary studies to delineate the best approach for ultimate process development. They included cursory experiments with physical separation of contained metallized particles, chemical leaching, and pyrometallurgical processing. The results of the preliminary experiments indicated that the pyrometallurgical approach would offer greatest possibilities for commercial incorporation into existing plants where the secondary alloy products could be recycled directly to the primary alloying operations.

Pyrometallurgical investigations included pelletizing furnace feed mixtures, followed by smelting and reduction tests using batches of up to 25 pounds.

A process was successfully demonstrated at the laboratory scale that resulted in recoveries of up to 95 percent of the chromium and 100 percent of the nickel and iron in the wastes. The alloy products contained 10 to 20 percent chromium, 6 to 12 percent nickel, and 55 to 65 percent iron, with lesser amounts of manganese and molybdenum. Lead and zinc, if present in the wastes, were recovered in a marketable oxide fume containing about 40 percent zinc and 20 percent lead. One commercial producer of stainless steel placed the value of a typical laboratory ingot at $385 per ton at 1973 prices.

The furnace slags, being very hard, friable, and free of toxic metals, should find a ready market as aggregate material.

Pollution Control and Waste Disposal

Since a relatively small amount of the wastes generated in the bulk raw materials processing sector are suited for further economic processing, large tonnages of these collected materials must be disposed of by nonpolluting methods. The processing plants in this sector must also avoid polluting any part of the environment by releasing solid, liquid, or gaseous wastes into the environment. Considerable research has been directed toward devising better ways to collect such wastes within the processing system and also to find suitable disposal methods.

Ultrasonic Flocculation of Metallurgical Smokes and Fumes

The well-known electrostatic precipitator is the most widely used device for removing fine suspended matter from smokes and other gas streams before they are released to the atmosphere. In operation, the electrostatic precipitator charges the suspended particles in a very high-voltage electric field so that a force is developed between the charged particles and one element of the precipitator. The force is such that the particles are moved to the collector element from which they can be removed periodically. The net result is a cleaner gas stream for release or other use, and collected particles for disposal or other use.

Although the electrostatic precipitator is used extensively, it is not suitable for many applications where the high operating voltage cannot be tolerated. The ultrasonic flocculator would be better suited for such applications if it could be operated economically on a commercial scale. It is based on the principles commonly demonstrated in classrooms with the classic Kundt tube in which a standing sound-wave field is generated within a cylindrical glass tube by stroking a metal rod attachment at one end. Cork dust, placed inside the tube, collects at evenly spaced intervals (half-wavelengths) along the length of the tube.

Gottschalk and St. Clair (22) first began to investigate the application of sound waves for flocculating smoke and fume in 1937. By 1940, St. Clair (74) had developed an electromagnetic sound generator that appeared to hold promise for many special commercial applications. St. Clair, Spendlove, and Potter (75) reported on the flocculation of aerosols by intense high-frequency sound in 1948.

The sound for these investigations (St. Clair's generator) was produced by electromagnetically exciting a solid metal cylinder of specific dimensions to vibrate at high amplitude in a resonate mode. A metal cylinder of duralumin or other alloy having low internal damping was excited into longitudinal vibration about a midsection-supporting web mounted on rubber cushions. The exciting force was induced into a hollow cylindrical cup or ring machined at one end of the cylinder. The cylinder was mounted so that the ring was evenly spaced to vibrate freely in the circular ring-gap of a pot magnet. The vibrational forces were essentially identical to those operating in the driving elements of a common dynamic loudspeaker. A thin cone or other diaphragm is the vibrating element of the speaker; the solid cylindrical rod is the vibrating element of the sound generator. Resonant frequencies are avoided in the speaker and promoted in the sound generator.

The high-frequency driving current was supplied from an electronic amplifier to feed a stationary coil wound on the center pole of the pot magnet. The driving ring on the end of the vibrator acted as a one-turn secondary of an electrical transformer. Strong electrical currents induced into this driving ring caused a magnetic field, which reacted with a constant magnetic field in the pot magnet gap, thus exciting the cylinder into vibration at its natural frequency. A flat plate of a condenser-microphone was placed against the center pole of the magnet. The end of the metal cylinder, inside of the driving ring, served as the other plate of the microphone pickup. The microphone was made to generate an electrical signal of a frequency and waveform corresponding to the vibrations of the metal cylinder. This pickup signal was shifted in phase, amplified electronically, then fed to the pot-magnet drive coil. This type of feedback was suitable for maintaining the cylinder in continuous vibrations at or very near its resonant frequency and at a high amplitude as long as the amplifier power was supplied. With metal cylinders of appropriate materials and dimensions, high-frequency, high-intensity sound could be radiated from the open end of the cylinders.

The aerosol flocculator equipment was comprised of a single cylindrical enclosure mounted vertically with the sound generator at the top and a movable reflector plate at the bottom (fig. 9). The sound generator radiated sound downward, and the reflector plate was adjusted to an exact wavelength distance so as to reflect the sound wave back onto itself and cause a standing wave sound field within the enclosure, commonly known as a tuned system. Standing wave sound fields of only 4 or 5 wavelengths had sufficient energy density to levitate coins or small glass spheres at the antinodes. The frequency of some generators were well within audible limits; several were far above. The intensity was such that earplugs were used for protection. Flocculation tests were conducted with various smokes and fumes generated in the laboratory under controlled conditions. Flocculation performance was determined by comparing

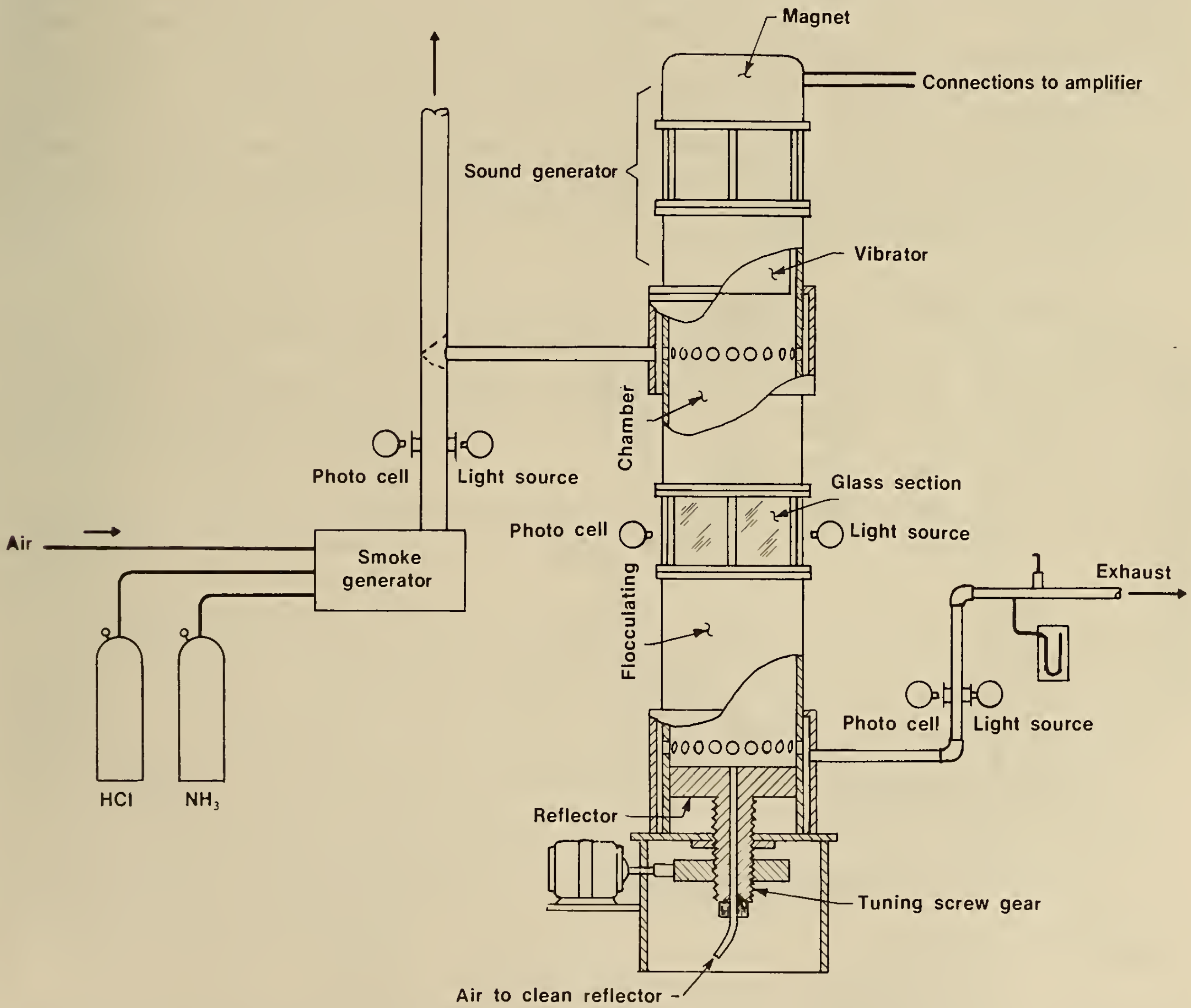

FIGURE 9. - Laboratory model of sonic flocculator (75).

the extinction of a light beam passing through unflocculated smoke with the light extinction of the same beam after flocculation. By using photoelectric cells to measure the light extinction and applying a modification of the Lambert-Beers law, it was possible to calculate a flocculation index for smokes of various weight densities and corresponding driving currents or power inputs.

The experiments included preliminary tests to determine the vibrator efficiency, flocculation chamber characteristics, and calibration of the smoke generator and the photocell equipment, usually with ammonium chloride aerosols.

Figure 10 is a typical representation of several flocculation tests. The rate of flocculation increased rapidly with sound intensity and time and was more rapid with the denser aerosols. In the frequency range from 4.9 to 8.8 kilocycles, no essential difference was noted, especially for aerosol densities of 4 to 5 milligrams of solids per liter. Calculations were also made to show that it is the radiation pressure in the sound field that concentrates or flocculates aerosol particles at the antinodes. Although ultrasonic flocculation was successfully demonstrated for a wide variety of conditions, economic commercial-scale application was not demonstrated. The high cost of very powerful electronic equipment, at that time, indicated no potential for commercial-scale application. The phenomenal advances made in electronic technology and devices since then would justify a new examination of present potentials for sonic flocculation.

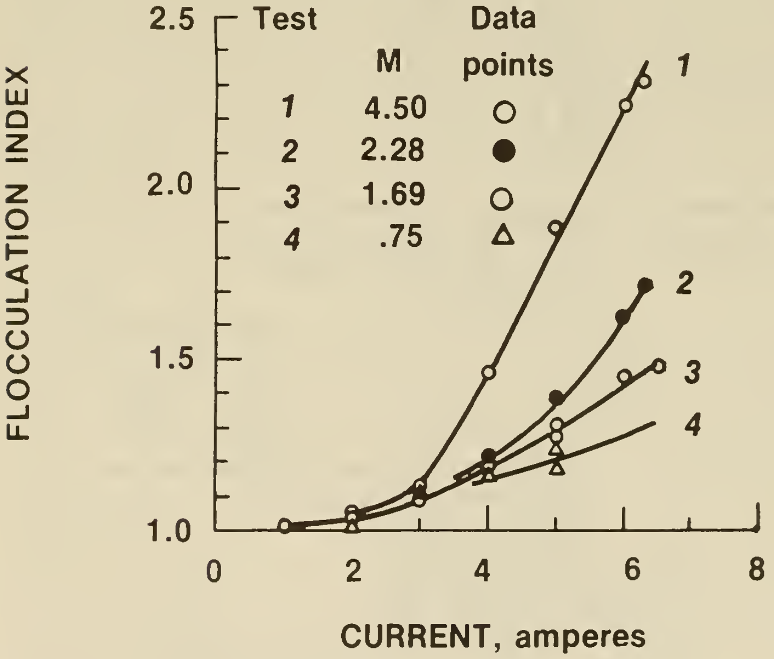

FIGURE 10. - Representation of sonic flocculation tests (M is weight of smoke particles per unit volume) (75).

Subsurface Disposal of Pickle Liquor

A common practice in the steel industry is to chemically remove the scale of iron oxides (FeO, Fe_2O_3, and Fe_3O_4) from the surface of steel strips, plates, and bars before final processing to finished products (7). The oxides are dissolved by subjecting the steel items to a bath of acid solution, a process known as pickling. Hydrochloric acid solution, for example, dissolves the oxides of iron to form ferrous salt. The effluent solution from the pickling vats contains a mixture of iron salts, residual acid and water. The average effluent from hydrochloric acid pickling solution contains about 18 percent iron chloride, and about 0.9 percent hydrochloric acid. Sulfuric acid pickling solution is also used but it reacts differently. It attacks the steel surface below the oxide mill scale, and the liberation of hydrogen forces the scale away from the steel surface. The waste pickle liquor (WPL) from sulfuric acid vats contains 14.3 percent iron sulfate and 9.8 percent sulfuric acid.

An estimated 11 million tons of steel products were treated by hydrochloric acid pickling in 1966. At a proportional rate, the estimated amount of steel products that will be treated in 1985 will be 15 to 20 million tons.[5] Obviously, tremendous quantities of WPL are generated each year, and disposal problems are increasing with the more stringent disposal regulations imposed in many areas.

A subsurface disposal technique was evaluated by Bayazeed and Donaldson (7) who reported on their results in 1973. Disposal of WPL into subsurface rock strata (sandstone) had been in practice since 1964 in Illinois, Indiana, Ohio, Pennsylvania, and New York. The report covered only the north-central Illinois and northwestern Indiana region where wastes were being injected into Cambrian Age, Simon formations, which had an average porosity of 12 to 14 percent and an absolute permeability ranging from 75 to 400 millidarcys. Permeability is a measure of the ability of a porous medium to transmit fluids. The Mt. Simon formations range up to 1,800 feet thick, and the average depth of WPL injection wells in the area was 4,000 feet. These wells were fed mostly by gravity flow at rates up to 4,000 barrels per day.

The investigations included studies on the design criteria for waste-injection wells, measurement of porosity and permeability of selected sandstone core samples taken at various sites, and simulated continuous injection of various solutions into typical core samples. Other studies compared the relative cost of injection disposal with WPL treatment at the surface and surface disposal.

The fact that WPL had been successfully injected into a sandstone formation for nearly 10 years was an adequate demonstration of practical feasibility.

Laboratory results indicated that most of the WPL acid would be neutralized and the iron absorbed irreversibly by the geologic formation after a long period of storage. The environmental impact of the method, even though widespread, would thus appear to be considerably less in the areas considered than discharging the WPL into a stream or landfill. It was also determined that the injection disposal method was three to six times less expensive than surface treatment and disposal. The loss of iron in the WPL is one negative aspect of the subsurface disposal method. Nearly 5 million gallons of WPL was then being injected annually. Assuming the average WPL density is 1.2 and contains 18.1 percent ferrous chloride, the amount of iron loss would exceed 10,370 tons per year.

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[5] This projection is based on table 14 of the Iron and Steel chapter of Mineral Facts and Problems (U.S. Bureau of Mines, Bulletin 667, 1976, p. 575).

Evaluating Rock Formations for Nuclear Waste Disposal

Studies similar to those described in the previous discussions were conducted by Champlin, Thomas, and Brownlow (18) to evaluate the suitability of various geologic strata for the disposal of nuclear wastes. The problem evolved with the rapid development of the nuclear materials reprocessing and power generation industries. Both industries produce radioactive wastes that must be totally and permanently contained to prevent the spread of highly toxic and dangerously radioactive wastes. The early high-cost, tank disposal practice was necessarily adopted without the benefit of preliminary long-term evaluation.

The type of waste is determined largely by its origin. Fuel elements, for example, differ in composition and cladding materials, matrix materials, and composition of spent fuel. The unused fuel is recovered from the different types of fuel elements by different techniques. Acid processing generates liquid acid wastes that contain various organic and inorganic substances that have been added to the process.

Following are some methods most seriously considered or widely used for the disposal of radioactive wastes: (1) Disposal of packaged wastes in the ocean; (2) adsorption, fixation, or fusing the waste materials in cements, ceramics, or glass, and subsequent disposal in pits, dry mines, or caverns; (3) disposal in shale beds by pumping a slurry of cement and wastes into deep-well shale strata; (4) disposal of packaged wastes in caverns cut into deep salt formations; and (5) injection of liquid wastes into permeable subsurface geologic formations.

The latter method was the subject of a 1959 cooperative study by the Bureau of Mines and the Division of Reactor Development and Technology, Atomic Energy Commission. The possibilities of particular interest included the injection of aged, nonboiling, highly radioactive wastes into rock formations of suitable porosity and permeability. One of the first tasks was to obtain samples of the rock formations from potential disposal sites and determine their respective porosity and permeability by standard laboratory techniques. Most of the samples tested were of the sandstone type.

Subsequent tests were conducted with a special suite of solutions including brine to measure their various reactions with the rock samples, their solution retention properties, and the ultimate suitability for disposal of specific types of wastes--acidic, alkaline, or brines.

The laboratory experiments indicated the feasibility of satisfactory injection and permanent disposal of liquid nuclear wastes in many of the natural geologic formations. By carefully monitoring and controlling the ionic balance and particle-size distribution in the waste, injected waste could be contained over a long period. Sandstone samples were shown to exhibit the best combination of permeability and porosity, with low cation-retention capacity. It was also found that shale and clay formations would make excellent confining materials because of their very low permeability and high cation-retention capacity. The experiments proved that rock cores could be used to provide good models of storage reservoirs for the laboratory determination of parameters essential for full-scale subsurface disposal of radioactive wastes.

MANUFACTURING AND SERVICES

Thousands of different commodities of mineral origin are consumed by the manufacturing industries each year. In 1974, the manufacturing industries consumed preprocessed commodities such as fuels, electricity, steel, aluminum, other metals, tile, glass, cement, chemicals, plastics, fertilizers, and others valued at $210 billion (97). The 1974 output of goods and services from this sector of the economy amounted to $1,397 billion, the gross national product. However, since the United States imports of mineral materials ($42 billion) exceed the exports ($18 billion), the 1974 minerals deficit amounted to $24 billion. This was a threefold increase from the 1973 deficit of $8 billion. By the end of 1975 the mineral deficit had been reduced only to $22 billion. Obviously, any improvement in manufacturing, utilization of materials, and recycling process would, in effect, increase domestic supplies and conserve domestic resources. Although the Bureau of Mines has investigated possibilities of improving many of the common materials-utilization and recycling operations in the manufacturing industries, there are still literally thousands of wastes yet to be identified and characterized for subsequent studies on recovery or disposal processes. The following discussions pertain to research activities that typify Bureau of Mines resource recovery research in the manufacturing sector of the supply-utilization system.

Recycling Scrap Titanium Alloys

The use of titanium for making special alloys for some goods and devices is relatively new in the manufacturing industry, beginning some time in the midfifties and growing to significant proportions by the midsixties. Currently, large amounts of titanium waste and scrap are generated at nearly all phases of the manufacturing operations. Although large amounts of in-plant alloys are recycled, the problem of handling contaminated scrap has become more complex. Titanium alloys were always adversely affected by very minor amounts of impurities so that the recycling of process scrap had to be practiced with ever-increasing caution. As more titanium alloys were used in conjunction with more of the other nontitanium alloys, the risk of contamination grew to serious proportions. The obvious approach to the problem of successfully recycling all scraps was to develop scrap-processing methods that would separate all contaminating materials from the titanium scrap rapidly and completely. An investigation of this approach was reported by Leone and Couch in 1974 (43).

In common practice, the titanium process sponge is mixed with clean titanium alloy scrap to produce new ingot. Unless rigorous procedures are followed in the collection and segregation of in-plant or purchased scrap, it cannot be recycled; however, the problem of segregating all types of scrap is very difficult. The problem involves the separation of alloys such as Ti-5Al-2.5Sn from other alloys such as Ti-6Al-4V. Another problem involves the removal of chromium-bearing stainless steel alloy scraps that may become mixed with the titanium alloys, and the removal of tungsten-carbide chips that break away from cutting tools during machining operations.

The objective of the investigation was to develop a processing unit and demonstrate a method for completely cleaning all types of contaminated titanium alloy scrap (chips and turnings) so that they would be suitable for reuse. The laboratory investigations included (1) solvent cleaning with perchlorethylene to remove heavy cutting oils; (2) detergent cleaning with a commercial alkaline cleaner; (3) anodic solution removal of impurities such as stainless steel 3.6, Hastelloy C, Inconel, and tungsten carbide; (4) chemical solution of the titanium scrap with nitric-hydrofluoric acid solution; and (5) physical separation after hydriding to render the titanium alloys friable so that they could be recovered selectively by crushing and screening, and then dehydriding to render them suitable for remelting.

The process ultimately developed included the following unit operations, all or any one of which could be employed by processing plants according to the type of waste materials available: (1) Solvent degreasing of contaminated feed scrap; (2) magnetic removal of iron and tungsten carbide; (3) aqueous hydroxide removal of aluminum alloys; (4) anode treatment in sulfuric acid to remove stainless steels, nickel alloys, etc.; (5) pickling in a nitric-hydrofluoric acid bath to remove surface oxides; and (6) washing and drying of processed scrap.

Ingots made with titanium scrap cleaned by the foregoing procedures were found to meet all of the rigid specifications for primary ingot. It was believed that steps 3 and 5 would not be necessary at most reclamation plants.

Nickel and Zinc From Waste Phosphate Solution

Many manufacturers of steel products follow a widespread practice of coating new steel surfaces with phosphate to provide a surface structure to which paints, enamels, or other coatings can adhere. Such surfaces also absorb oils so that, when necessary, they will provide the lubrication in die-forming operations. The coating solutions are comprised of phosphoric acid and metal phosphate compounds and when spent, they must be replenished. Disposal of the spent solution (waste) has caused a growing pollution problem in many locations, and a serious loss of phosphate and metals. The problem of recovering valuable constituents from waste phosphate solutions was investigated by Powell, Smith, and Cochran (66).

If it is assumed that 20 percent of the thin sheet steel produced annually in the United States is phosphate coated, the amount of spent phosphate solution that would be generated each year is on the order of several million gallons. These wastes have been dumped into sewers, rivers, and streams for years. The contained phosphate stimulates the undesirable growth of algae and other plants in the rivers, lakes, and streams. These wastes also contain metals that can be toxic to the fish in the water and livestock that may drink it.

The objective of the investigation was to evaluate the solvent extraction technique for selectively recovering nickel and zinc from the waste solution commonly generated during the phosphating of automobile bodies. The investigations were to be a preliminary effort with the ultimate hope of recovering values from the solid phosphate sludges. They contain more valuable constituents, and in greater quantities than the solutions, but are more difficult to process.

New phosphate solution is essentially a mixture of phosphoric acid, a primary phosphate of either iron, zinc, or manganese, and oxidizing agents such as peroxides, nitrates, nitrites, or chlorates. The coating action stems from the insolubility of most metal phosphates in water. When applied to clean steel surfaces the solution dissolves iron and releases hydrogen. The primary metal phosphates are converted to insoluble secondary and tertiary phosphates that adhere strongly to the steel surface. Because the presence of the evolved hydrogen slows the phosphating reaction, the oxidizing agents are added to convert the hydrogen to water and thereby sustain rapid phosphating.

The untreated surface of most steels is susceptable to corrosion and is often relatively rough. The phosphated surface is relatively much smoother and corrosion resistant. It is strongly adherent and forms an excellent surface to receive paints, enamels, or other liquid coating materials. The composition of most phosphating solutions are usually proprietary.

After a series of laboratory experiments, a solvent extraction process was developed which included preliminary zinc extraction, phase separation, and zinc stripping, and a similar circuit for nickel. It was found that the recovered zinc, nickel, and phosphate products were essentially uncontaminated by each other. At least 99 percent of the zinc was extracted by di-2-ethylhexyl phosphoric acid in kerosine, and 99 percent of the nickel was extracted by dinonyl napthalene sulfonic acid in butyl ether. Both extractions were higher when zinc was extracted first. Nearly all of the phosphate remained in the aqueous raffinate. It was possible to strip each organic phase of its metal load with sulfuric acid. The stripped organic was then ready for recycling.

Although both zinc and nickel could be almost completely recovered and easily stripped, subsequent treatment would be necessary to produce marketable products. It was also found that 30 percent of the sodium in the waste was recovered with the nickel, and the balance with the phosphate.

It appeared that a process of this type would be suitable for treating other similar waste solutions containing nickel and zinc.

Processing Wastes With Wastes

One of the most unique resource recovery processes was developed by Cochran and George (19) for the reclamation of valuable constituents from a number of electroplating and etching wastes. The unique feature of the process involves the mutual treatment of two types of wastes with each other and the recovery of the valuable constituents of both.

Because no previous recovery technology had been developed for wastes of this type, large tonnages of the high-value metals contained in electroplating solutions and other similar industrial wastes had been discharged for years into sewers, rivers, and streams. Earlier proposed methods either failed to recover all values, produced additional troublesome wastes, or were too expensive to be practical.

At an estimated nickel-plating efficiency of 80 percent, the amount of nickel lost in electroplating wastes was 5,000 tons in 1970. The estimated annual loss of metals in several types of wastes generated at two typical electroplating companies is given in table 4. There were approximately 20,000 electroplating companies in operation at the time of the foregoing analyses.

TABLE 4. - Metal losses at two typical electroplating companies (19)

Type of waste	Firm	Metals lost, short tons[1]					
		Ag	Co	Cr	Cu	Fe	Ni
HNO_3	A	-	0.08	-	2.9	0.1	6.2
CN	A	-	-	-	3.4	1.9	1.2
CN	B	0.37	-	-	.2	<.1	<.1
Cr	B	-	-	0.6	.9	<.1	<.1
Total	-	.37	.08	.6	7.4	2.0	7.4

[1] Based on average of 3 to 10 samples of rack-stripping and chrome-acid-etching waste solutions.

The common electroplating practice involves placing the objects or pieces to be plated on metal racks, which in effect connects them all together to form the cathode of the plating cell. The electroplating process not only plates the objects with metal but also the racks so that they must be stripped with nitric acid periodically to remove excess metal, a process that generates nitric acid rack-stripping waste solutions. Another similar industrial waste solution is generated during the chromic acid etching of printed circuit boards for the electronics industry. Still another kind of rack-stripping solution is the alkaline-cyanide type, which contains sodium cyanide and sodium m-nitrobenzene-sulfonate. The composition of common waste solution samples from three different industrial firms is shown in table 5.

Cochran and George (19) reasoned that one very effective first step for processing these acid and alkaline wastes would be to neutralize one with the other under very carefully controlled conditions. It was stressed that such a procedure would be highly dangerous if not properly performed. For example, if the cyanide were added to the acid waste, or if too much acid were added to waste solutions containing high concentrations of cyanide (CN), or even if the acid were added too rapidly, dangerous amounts of the highly poisonous hydrogen cyanide gas (HCN) would be evolved. The sequence and control of additions were thus of critical importance.

TABLE 5. - Composition of concentrated waste solutions, grams per liter (19)

Type of waste	Firm	Free CN	Ag	Co	Cr	Cu	Fe	Na	Ni
HNO_3	A	-	-	1.3	-	49	2.1	-	190
HNO_3	A	-	0.001	6.7	-	65	3.1	-	97
Cr	B	-	-	-	51	52	-	1.3	.01
Cr	B	-	-	-	30	56	.06	-	.003
Cr	B	-	-	-	28	57	.06	-	.003
Cr	C	-	-	.004	43	.06	.05	.07	.015
CN	A	90	-	-	-	19	15	-	5.1
CN	A	52	-	-	-	19	.72	98	5.5
CN	B	62	2.2	-	-	8.6	<.01	83	4.6
CN	B	113	.9	-	-	7.7	.06	75	.01
CN	B	94	5.5	-	-	5.9	<.01	105	1.4
CN	B	26	28.9	-	-	1.4	.20	17	.01

A practical procedure was ultimately found to handle these wastes as mutually effective processing reagents and with uncommonly high recoveries of all contained values. The approach was to add the acid wastes to the strongly alkaline CN wastes under carefully controlled conditions to neutralize both wastes and precipitate the resulting metal cyanides. Several alternative procedures were studied in depth.

It was found that these types of waste can, in fact, be confined without difficulty under controlled conditions and that the metals and cyanides were almost completely precipitated. The process was successfully demonstrated with both concentrated and dilute waste solutions of the major electroplating and etching types for the recovery of Cd, Cr, Cu, Ni, and Zn. It was particularly attractive from an economic point of view when applied to concentrated electroplating, anodizing, and etching waste solutions primarily because of the very low operating cost and the simplicity of mixing the two wastes. Filtrates from the first or combining step of the "waste-plus-waste" process were considerably less dangerous to handle than the unprocessed wastes. The filtrates met the Public Health Service standards for free cyanide. At a detection limit of 0.03 ppm, no free cyanide ions were detected in most of the filtrates. The amount of hydrogen cyanide produced during the neutralizing step was small in quantity and easily collected and neutralized.

Silver From Photographic Solutions

A 1968 report by Dannenburg and Potter (22) describes a simple but effective method for recovering the silver content of spent photographic solutions. They reported that the 1965 domestic consumption of silver for industrial and artistic purposes amounted to 137 million troy ounces, but only 40 million ounces of the total demand was supplied from domestic mines. During the same year, the photographic industry consumed 45 million ounces (33 percent) of the total demand. About 20 percent of the silver used in photography was for image formation on the films or papers. The remaining 80 percent was dissolved by the thiosulfate fixing bath known as "hypo" solution.

A very simple method was developed for recovering the silver normally lost in spent hypo solutions. Most of the larger commercial photographic processors and medical photography facilities have installed some form of silver-recovery units, generally of the electrolytic type, which are usually expensive to install and operate. The investigation was aimed at the design and evaluation of an efficient but simple and economic metallic displacement process for recovering silver from waste photographic fixing solution. A unit was developed in which easily available and cheap steel wool or steel window screen was packaged in a convenient container of appropriate size for recovery at any scale. It was well adapted to supplement the electrolytic-type recovery systems in use. A smelting process for the recovery of pure silver from the precipitated (sludge) silver was also developed and tested.

Preliminary calculations were made to estimate the potential for silver recovery from photographic wastes. It was found that 1 gallon of fixing bath solution from X-ray film processors contained about 1.2 troy ounces of silver; most other film fixing baths had 0.4 to 0.6 ounce. The paper-fixing solutions had 0.05 to 0.3 ounce per gallon of silver. Considering the amount of paper or film processed, it was estimated that 1 troy ounce of silver could be recovered from the hypo solution generated during the processing of 8,000 square inches of X-ray film, 16,000 square inches of other black and white film, or 64,000 square inches of black and white paper.

The photographic fixing solutions, which remove unreduced silver halides from the developed emulsions, contain sodium or ammonium thiosulfate, which readily dissolves silver halides but does not dissolve metallic silver. Other constituents of the fixing solutions include varying amounts of sodium sulfite, acetic acid, boric acid, and chrome or potassium alum.

The constituents of several typical spent fixing baths are shown in table 6.

TABLE 6. - Composition of typical spent photographic fixing baths, grams per liter (22)

Type of solution	Ag	Na_2O	K_2O	Al_2O_3	NH_4	$COOH^-$	$S_2O_3^=$	$SO_3^=$
Aerial print fixer...............	0.65	21.8	0.7	0.9	-	6.0	33.9	2.16
X-ray fixer......................	10.1	14.6	3.1	2.8	31.9	28.0	91.4	8.8
Black and white fixer prints......	1.0	(1)	(1)	.2	-	1.5	66.6	13.0
Black and white fixer film........	5.6	(1)	(1)	1.8	-	67.0	121.0	9.1
Color print fixer................	1.56	(1)	(1)	.9	-	(1)	24.3	13.5
Color film fixer.................	3.76	(1)	(1)	.2	-	1.0	91.8	8.4

[1] No data.

To standardize all experiments and evaluations more accurately, a standard synthetic hypo solution was prepared as a stock for all experiments. The constituents, in grams per liter, included sodium thiosulfate, pentahydrate, 240; sodium sulfite (anhydrous), 15; glacial acetic acid, 13.1; boric acid, 7.5; and potassium alum, 15.

Laboratory evaluations included studies on the following: Precipitation rates with different replacement metals; different physical forms; the effects of barren hypo on the metallic precipitants; the effects of retention time, hypo acidity, and thiosulfate concentration; and smelting of the sludges by both reduction and oxidation techniques.

The results indicated that the iron filament type of replacement metal in the form of 00 grade steel wool was ideal for precipitating the silver as a high-grade sludge from acidic waste photographic fixing solutions containing from 1 to more than 10 grams of silver per liter. A smelting process demonstrated that pure silver could be smelted from sludges containing 27 to 80 percent silver. The apparatus for the precipitation operation was simple to construct, inexpensive to build and operate, and performed efficiently at any scale.

SECONDARY MATERIALS INDUSTRY

The secondary materials industry has been a relatively small but well-established part of the materials supply system for hundreds of years. Today the industry supplies a substantial part of the total U.S. consumption of many metals, glass, paper, fabrics, and others. Without the secondary materials industry, millions of tons of scrap metals and wastes, containing thousands of tons of aluminum, copper, zinc, lead, tin, iron, and other metals would have accumulated each year at waste dumps across the country. Such dumps would have also contained substantially greater quantities of glass, paper, textiles, rubber, and other important materials. Fortunately, the secondary materials industry has processed and recycled large amounts of these postconsumer materials saving the Nation untold billions of dollars in natural resources and energy over the years.

The importance of this industry is reflected by the productivity of the secondary-metals producers (97). This part of the U.S. recycling industry recovered metals from old scrap equivalent to about 35 percent of the lead consumed in the United States in 1974, more than 50 percent of the antimony, more than 25 percent of the silver, nearly 25 percent of the copper, and about 20 percent of the nickel and tin. This recycling industry also produced 25 percent of the 1974 iron consumption and significant amounts of other metals such as aluminum, zinc, chromium, nickel, mercury, tungsten, tantalum, cobalt, gold, and platinum-group metals. The segment of the industry that recycles nonferrous metals, paper, and textiles has an annual sales volume of $9.2 billion (57) and employs nearly 68,000 people with an annual payroll greater than one-half billion dollars.

Many of the current processes employed by this industry are efficient and nonpolluting, but some are not. The Bureau of Mines has traditionally included secondary materials processing and utilization problems in its research programs for more than 50 years. Although this type of research did not comprise a prominent part of the earlier programs, resource recovery research was increased significantly in scope and depth during the mid-1960's when the general concern for diminishing domestic mineral supplies and environmental pollution problems stimulated considerable public support for greater research efforts. Research on the problems attending secondary-materials processing and utilization is still a substantial part of the Bureau's overall research program. The number of publications on these research investigations have increased many fold, and the results indicate considerable success.

Recovery

Distillation of Metals

Distillation technology has been used successfully for separating volatile constituents from liquid solutions for centuries. The chemical engineer has long used the distillation technique as one of his most efficient methods for separating organic compounds. Metallurgists have also been able to separate some metals from each other by taking advantage of their different vapor pressures. However, this practice was not regarded as a practical metallurgical process until the fifties and sixties when great advancements were made in the field of vacuum technology. These developments prompted reexamination of many possible metallurgical applications for distillation processes, and the results have been largely positive. The Bureau of Mines programs included research on selective distillation as a technique that might be applied generally in separating and refining metals, particularly nonferrous secondary-metals scrap, drosses, and residues.

Results of preliminary experiments by Caldwell, Spendlove, and St. Clair (15), on the removal of volatile metals from lead and tin by vacuum distillation, showed that cadmium, zinc, and tellurium could be effectively removed from lead, but the tellurium was evaporated with lead in quantities approaching the composition of the intermetallic compound Pb_2Te. It was difficult to remove bismuth or antimony from either lead or tin at temperatures up to 1,000° C. It was also noted that tellurium was removed from tin as the intermetallic compound SnTe.

In other studies, it was found that zinc could be vacuum distilled from galvanizers' dross, zinc-base die-cast scrap, and zinc-aluminum alloys at much faster rates and much lower temperatures than could be accomplished by distilling at atmospheric pressure. The experimental results indicated that vacuum distillation may offer several important advantages over conventional methods of refining volatile metals. Among the most important are high rates of evaporation at relatively low temperatures, increased selectivity, and protection of the bath and condensed metal from oxidation. However, there were several disadvantages in the method, including the cost of the vacuum system and the need in most vacuum furnaces to use electrical induction heating. Fuel-fired vacuum retorts had not been used extensively because of difficulties encountered in the construction of inexpensive gastight retorts having good thermal conductivity and corrosion resistance (80, 83).

A gas-fired distillation furnace was developed by Caldwell, St. Clair, Bilbrey, and Spendlove to circumvent the foregoing disadvantages of conventional induction-heated vacuum retorts (14). The retort was comprised of a graphite linear enclosed in a thin shell of stainless steel. The graphite provided the mechanical strength to resist the inward pressure of the atmosphere and served as a crucible to contain the metal. The stainless steel shell protected the graphite from oxidation and flame erosion and provided a gastight enclosure. Graphite was a satisfactory conductor of heat, particularly at lower temperatures; its thermal conductivity is considerably greater than stainless steel. One version of the various systems investigated is illustrated in figure 11. When the retort was used to distill zinc from galvanizers' dross and zinc-base die-cast scrap, more than 99 percent of the zinc was recovered in a water-cooled condenser as a solid condensate containing 99.8 to 99.9 percent zinc. Atmospheric distillation resulted in 95- to 98-percent zinc recovery at a purity of 99.5 percent. Distillation rates of 95 pounds of zinc per hour were attained at a temperature of 650° C with a 20- by 66-inch cylindrical retort containing 3,000 pounds of dross. Pressures ranged between 50 and 100 microns. Zinc distillation at this low temperature would not be practicable at atmospheric pressure. The retort was also found to be suitable for distilling other volatile metals such as cadmium and magnesium.

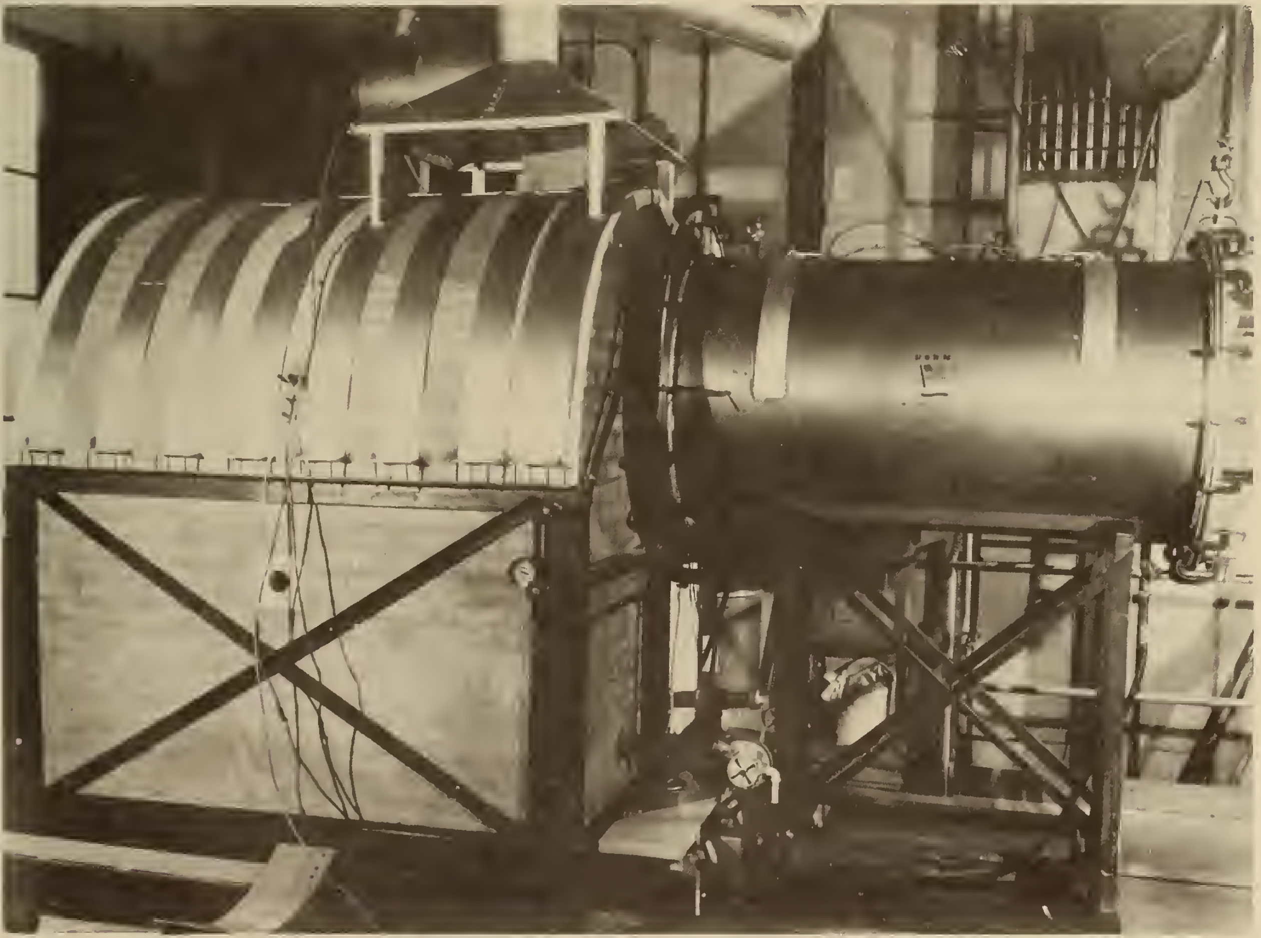


FIGURE 11. - Gas-fired vacuum distillation equipment (14).

Heat consumption using modern oil-fired zinc distillation equipment ranged from 7,300 to 8,000 Btu per pound of zinc distilled. The condenser in this case was air-cooled with sufficient thermal insulation to maintain a pool of condensate just a few degrees above the melting point. Observed average fuel requirements for vacuum distillation of zinc in the temperature range of 625° to 650° C was 9,250 Btu per pound of zinc distilled. However, this included heat used to raise the temperature of the furnace, retort, and charge from room temperature to 625° C. Only 6,500 Btu was consumed per pound of zinc distilled during the actual distillation period. These values show that heat requirements for vacuum distillation compared favorably with the performance of commercial atmospheric distillation units. It was assumed that still higher thermal efficiency could be achieved in full-scale vacuum retorts and furnaces.

The experimental vacuum retort was in service for 1,180 hours at distilling temperatures ranging from 570° to 650° C, which corresponds to furnace temperatures of 665° to 875° C, before repair on the stainless steel shell became necessary. The exterior surface of the retort showed no evidence of failure except in one area, which was under apparent strain where the shell had buckled away from the liner, probably because of improper fitting during construction. Repeated expansion and contraction due to the heating and cooling of the retort caused a 6-inch hairline crack to develop in the shell. However, repairs were made readily by welding a tight-fitting patch over the cracked area. At the time of the evaluation, the retort had been in use for 470 hours without additional repairs. Neither the patch weld nor the welded seam along the length of the retort had shown any sign of failure.

The graphite liner showed some evidence of wear but it was so slight that the length of additional service could not be predicted. Care in charging and cleaning the retort and protection from exposure to air while hot helped to keep the liner in good condition. The performance record for this retort indicated an initial service period of at least 1,000 hours after which minor repairs could probably extend its service life by several thousand additional hours.

In subsequent experiments, the gas-fired vacuum distillation equipment was modified to condense zinc in a liquid state rather than in a solid. Collectors and freeze valves were installed so that the condenser could be tapped of its liquid contents without opening the retort or condenser to atmospheric pressure.

Obtaining and holding a vacuum on a molten metal refining system adds significantly to the operating and capital costs of a distillation process, but the process offered higher rates of distillation at lower temperatures, which resulted in overall lower energy consumption.

Metallic Solid-Liquid Separations (Filtration)

Gravitational force is the usual means of effecting the separation of mixed solids and liquids having markedly different densities. A crust of solid-phase constituents can be skimmed from the surface of a molten metal pool if the solid phase has a lower density than the liquid pool. Conversely, the solid-phase constituents will sink to the bottom of the containing vessel if the solids are heavier than the liquid metals. The constituents are thus separated in the first case by skimming solids from the surface of the molten metal and in the second case by filtering the molten metal away from the solid residues. Such separations may be enhanced by forcing the separation at faster rates and higher levels of completion by the use of pressure, vacuum, or even centrifugal forces. All of these techniques were studied in a variety of applications for upgrading a series of selected binary alloys and waste materials generated in the production and utilization of the common nonferrous metals. The success of each experiment was determined by comparing laboratory results with theoretical calculations made with the aid of appropriate phase-diagram data.

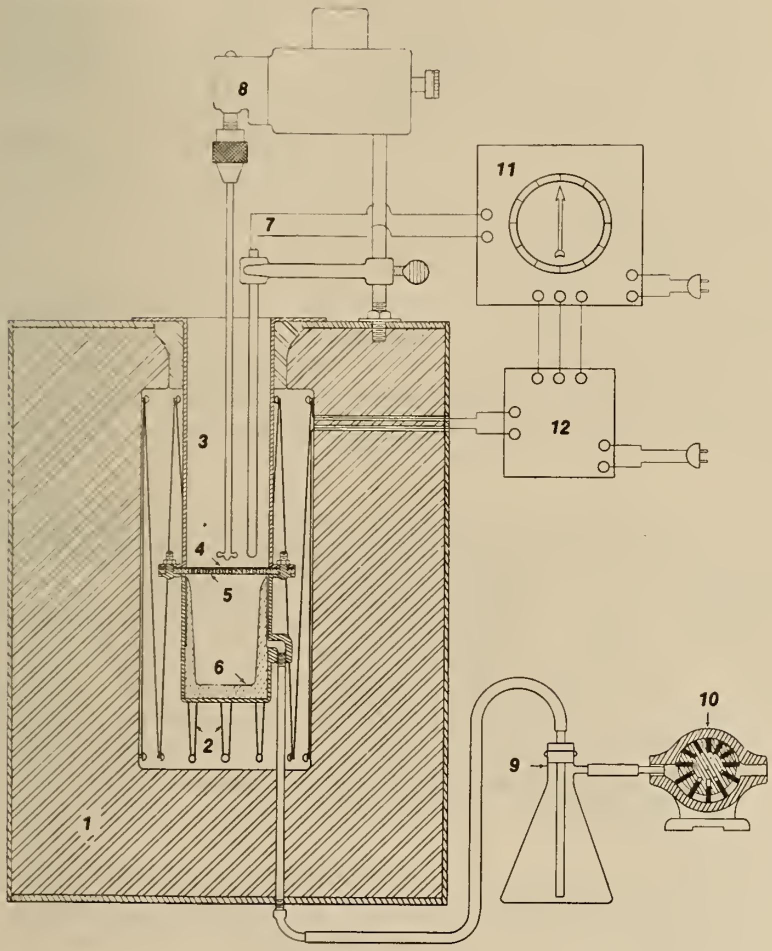


FIGURE 12. - Vacuum filtration of molten metals (73).

A simplified version of the filtration apparatus used by Ruppert and Sullivan (73) for separating the phases in binary molten alloys is illustrated in figure 12. The molten metals were heated to appropriate temperatures, then cooled gradually to promote the growth of solid-phase crystals. The molten system was contained in a vessel provided with a bottom filtering material of such porosity that the metal would not diffuse through it under its own hydrostatic pressure. After appropriate crystallization time, a vacuum was applied to the crucible causing the molten metal to flow down through the filter section leaving the solid-phase constituents on the filter bed.

A typical application involved the treatment of an industrial iron-zinc dross. A small amount of aluminum was added to the dross to form an iron-aluminum solid phase, which could be filtered away from liquid zinc as it was liberated from the iron. More than 98 percent of the zinc was recovered in most tests. The liquid phase (zinc) contained about 0.05 percent iron and as low as 0.13 percent aluminum in several tests.

Results from earlier experiments indicated that the several disadvantages of the vacuum filtration technique might be eliminated by the use of centrifugal force to produce clean liquid-solid separations. In the vacuum system, selection of the most efficient filtering material may be difficult for some alloys. The filter vessel might also leak prior to filtration or it may become clogged during filtration. Furthermore, the vacuum only added an additional 15 pounds per square inch to the force of gravity during filtration.

Schellinger and Spendlove (76) investigated the centrifugation technique using the system illustrated in figure 13. A spinner pot was built to operate in a furnace, and a collector channel was provided to catch the liquid-phase discharge. At the appropriate time, the pot was rotated up to several hundred revolutions per minute, thus exercising a strong centrifugal force on all materials in the pot. The solids were forced hard against the pot wall, but the liquid diffused through the solid phase and was forced out of the pot by the force of liquid metal accumulating against the inner wall. An example of separations achieved was demonstrated with a bismuth-30 percent lead binary alloy centrifuged at 165° C. The percent of theoretical liquid-phase metal recovered was 98 percent and only 9.5 percent of the solid-phase weight was occluded liquid. The centrifuge was found to provide excellent force for the separation, and in most alloy systems, the solid phases that collected against the pot wall were sufficiently coarse to allow the liquid to flow freely through the solid cake.

The major disadvantage of the batch centrifugation technique was the need to dismantle the equipment after treating each melt even though the collected solids often occupied a small fraction of the pot capacity. Another disadvantage would be the difficulty and expense to build and operate centrifugal equipment of very large capacity. Montagna and Ruppert (54) investigated the submersible centrifuge illustrated in figure 14 in an effort to overcome the disadvantages of the common batch centrifuge operation. It was intended that the submersible unit could be used to process very large vats of metal and need only to be withdrawn from the melt occasionally in order to dump out the collected solid phase.

In this application, a perforated cylindrical bowl was fastened to the end of a shaft which could be rotated rapidly and lowered or raised into and out of the metal bath. Blades in an opening at the bottom of the bowl pumped liquid into the bowl where the centrifugal action forced it horizontally through a filter fastened against the perforated wall of the bowl. The solid constituents collected on the filter, and the liquid discharged through the perforation and fell back into the bath. After sufficient time, all of the metal contents of a large vat had passed through the filter one or more times. The bowl was then raised and the collected solids discharged. After sufficient filtering, all of the metal in a large vat was free of solid-phase impurities. The results were comparable to those obtained by vacuum or pressure batch filtration of the same materials.

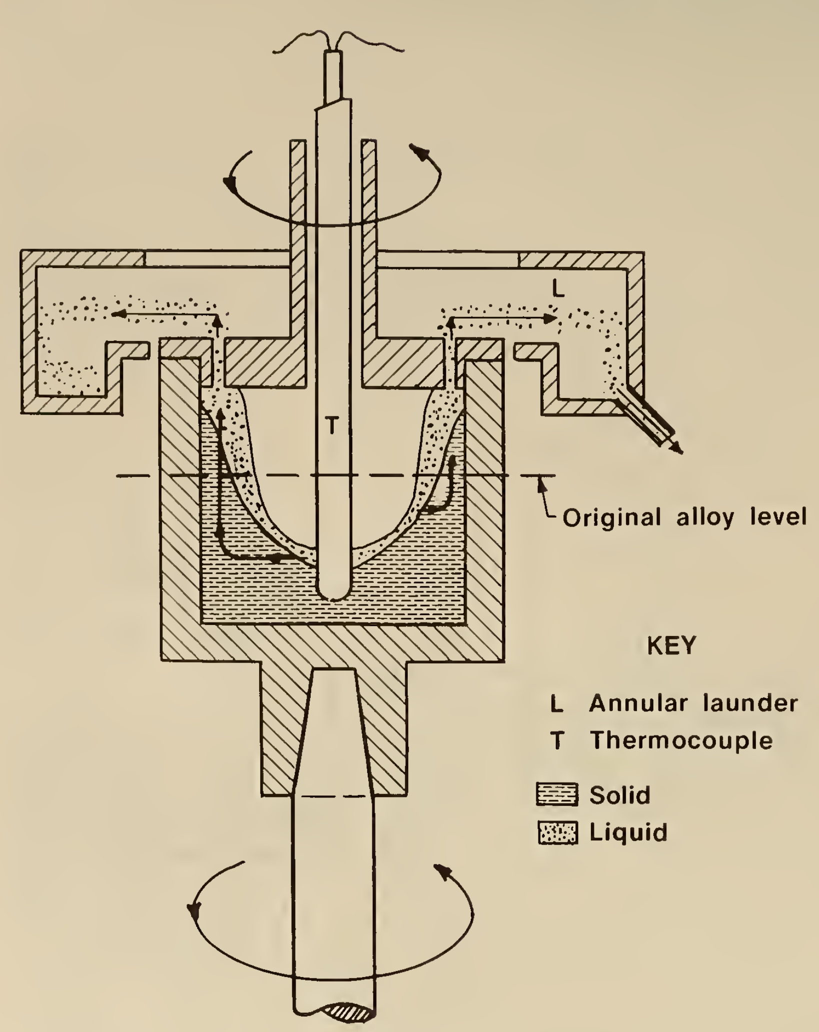
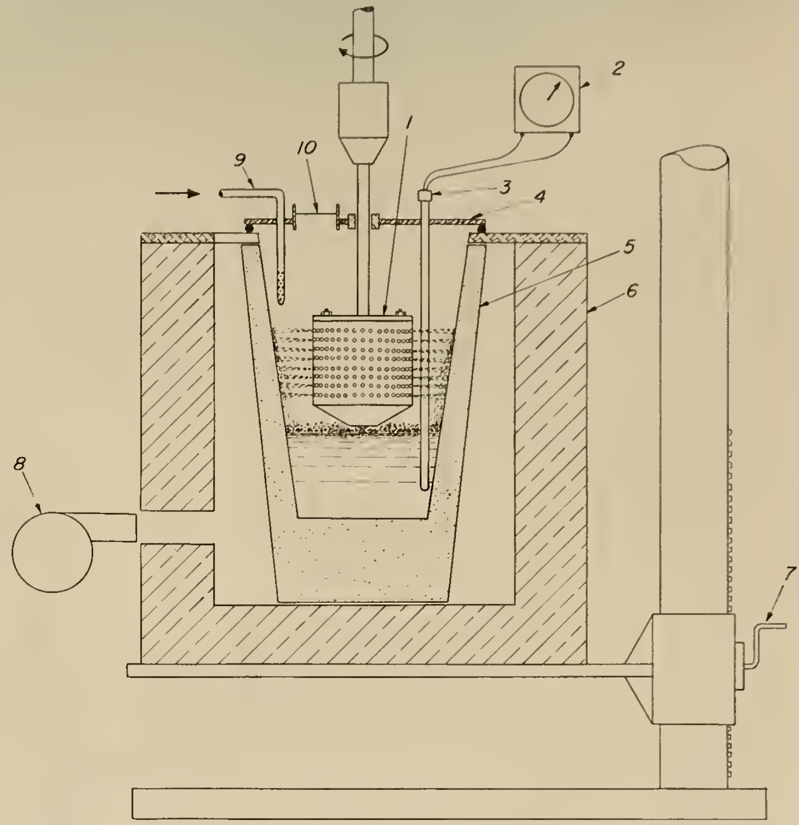


FIGURE 13. - Centrifugal filtration of molten metals (76).



KEY

1	Centrifuge bowl	6	Furnace
2	Indicating pyrometer	7	Elevating mechanism
3	Thermocouple	8	Gas burner
4	Crucible cover	9	Nitrogen inlet
5	Melting crucible	10	Sight glass

FIGURE 14. - Submersible centrifuge for molten metals (54).

Metal Separations by Preferential Melting

A large part of reclaimed metals is in the form of iron and copper components that are mechanically fastened to each other so as to be virtually inseparable. Devices such as electric transformers, generator parts, and electronic gear contain large amounts of copper and brass so closely associated with iron, steel, and other metals that melting is the only practical approach to separating these metals into individual products. The conventional "sweating" process is a relatively simple one in which the scrap is heated in the sweating furnace to the melting point of the lowest melting constituent. This fluid constituent then flows away from the solid scrap and collects in one section of the furnace. The conventional process of melting or "sweating" lower melting metals from those components that remain solid at sweating temperatures often leads to problems of extensive alloying and oxidation, thus markedly lowering the market value of recovered metals. In order to eliminate these difficulties, Leak, Fine, and Dolezal (41) developed several molten-salt bath preferential melting techniques using a variety of pretreatment procedures and various combinations of salt mixtures for the melting bath. The process, illustrated in figure 15, is straightforward and in some cases may be simplified still further by eliminating the pretreatment phase. Preliminary treatment may only be necessary for types of scrap that tend to oxidize or alloy excessively. Pretreatment with chemical reagents can alter and coat the metal surfaces, which in effect retards oxidation and forms an interface between solid and molten constituents to prevent intimate contact and alloying.

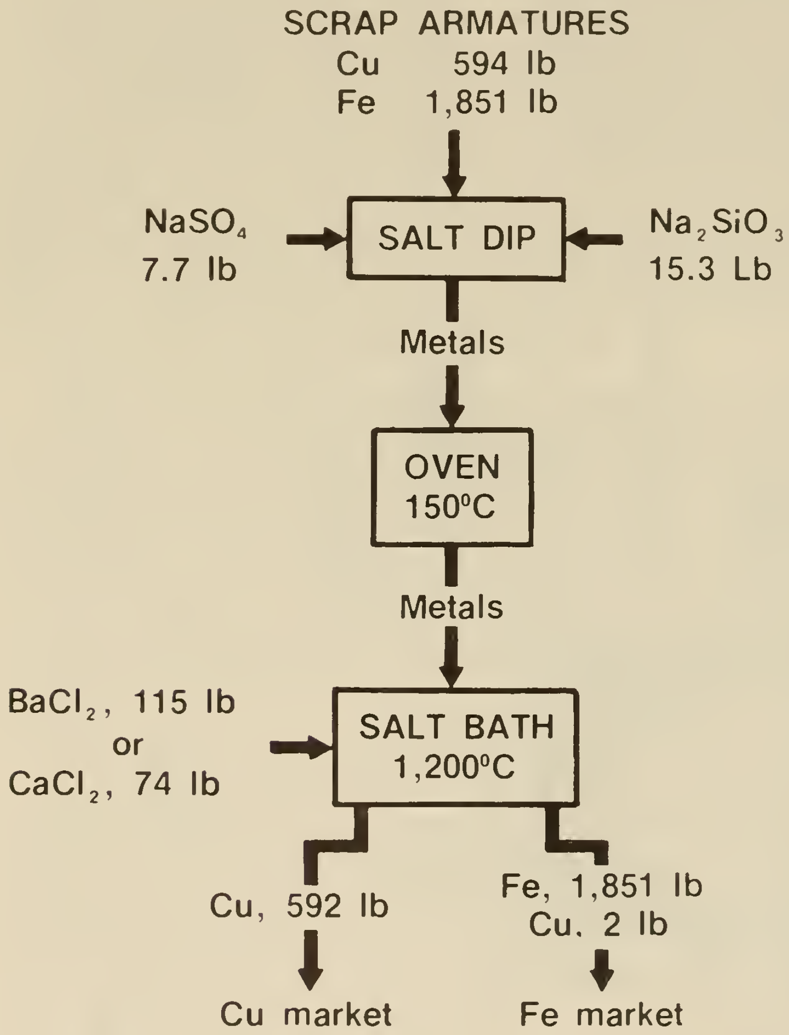


FIGURE 15. - Preferential melting of mixed metals and alloys (40).

It was found that by using a pretreatment procedure of either Na_2SiO_3 or Na_2SO_4, copper could be separated from iron in a barium chloride bath almost quantitatively. The data shown in figure 15 indicate a loss of only 0.3 percent of the original copper present. The process would be most efficient when the value of the melted constituent is sufficient to bear nearly all of the operating costs. Melting copper constituents away from iron components is an example of such a process.

Liquid Metal Alloying

Worn cutting and drilling bits comprise a very high-value scrap containing cobalt and cemented carbides. Although some of these high-value materials could be processed for recycling, the methods are either slow or expensive and only 70 percent of the contained hard constituents could be recovered as usable products. Barnard, Starliper, and Kenworthy (6) solved those problems by developing the simple and efficient process illustrated in figure 16. Studies showed that zinc readily alloyed with the cobalt in the scrap and the zinc-cobalt alloying caused an expansion of the scrap materials that loosened the carbide particles. The action was essentially a weakening of the microstructure due to alloying in an inert atmosphere. About 1 pound of high-grade zinc per pound of scrap was required, and alloying was complete in 2 hours at 800° C. The zinc was then recovered for recycling to the process by vacuum distillation at 800° C for 6 hours in a vacuum of 10 to 50 microns. The zinc condensate amounted to 99.9 percent of the original zinc addition. Distillation residues, when cooled to room temperature in a vacuum or under an inert atmosphere, formed a friable spongy mass. Essentially all of the carbides, cobalt, and unevaporated zinc remained in the residue. This residue was ground rather easily to produce a usable powder product containing particles of essentially the same size range (1/2 to 1 micrometer) used to make the original cemented materials. The process was equally effective on two different cemented-carbide scrap compositions. One contained 94 percent tungsten carbide particles cemented with 6 percent cobalt. The other

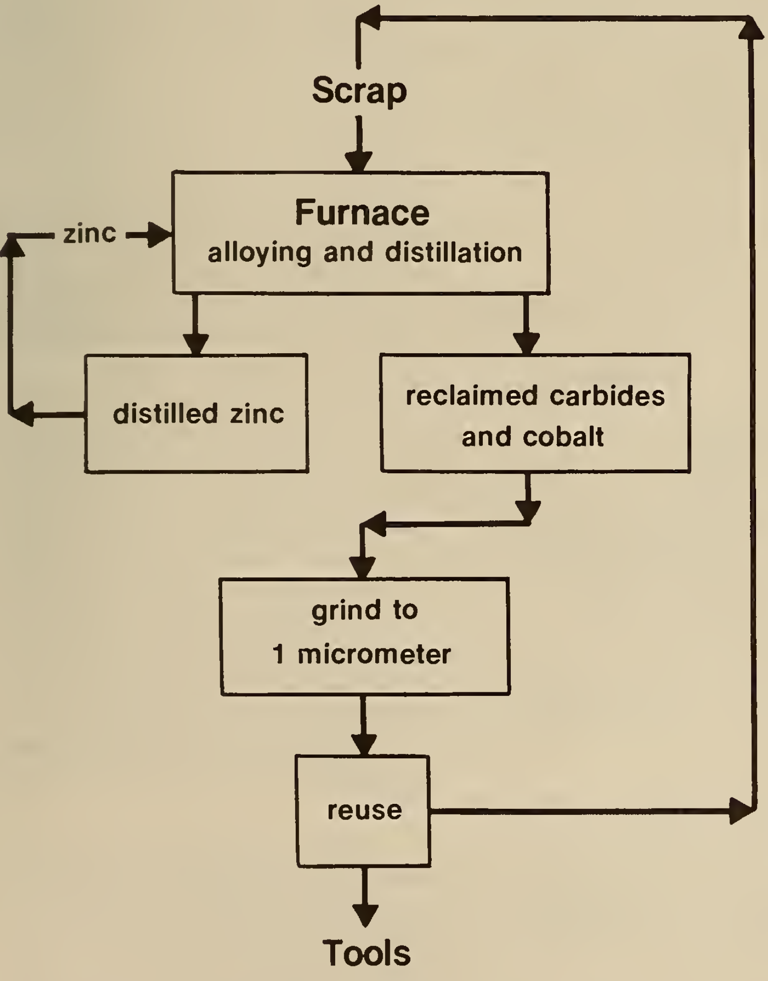


FIGURE 16. - Alloying and distillation separations (6).

contained 72 percent tungsten carbide, 8 percent titanium carbide, and 12 percent tantalum carbide particles cemented with 8 percent cobalt. The process is simple, cheap, and efficient. It produces high-purity products with no wastes, and the zinc reagent is recyclable.

Electrolytic Processing Using Amalgam Cells

The use of electrolytic methods for extracting the refining scrap metals has not received extensive consideration in the United States for several reasons. Perhaps the greatest problem is the wide variety of metal materials that are so difficult to accommodiate by conventional wet-chemical methods in order to produce satisfactory electrolytes and electrodeposited metal. The possible use of mercury as an amalgam electrode and as a vehicle to extract and transport metals rather selectively was investigated several years ago (46-47) in an effort to develop more efficient methods for processing tin and iron-bearing zinc alloys. Sullivan and Chambers (87-88) conducted experiments in which mercury was used in difficult processes to leach tin from hardhead (tin-iron compounds) and zinc from galvanizers' dross (zinc-iron compounds). The zinc and tin were subsequently electrodeposited respectively as high-purity metals. The zinc recovered from galvanizers' dross was 99.9 percent pure at an extraction efficiency of 98 percent. The tin recovered from hardhead was 99.99 percent pure at a recovery efficiency of 81 percent. The high-purity metals were produced by the same methods, but in separate systems. In subsequent research, a simple vertical amalgam electrode was developed to simplify the cell design and minimize the required mercury inventory. The experimental cell (fig. 17) was operated with the amalgam electrode in a vertical position. This was made possible by forming an amalgam on the surface of the anode metal so that the rich amalgam flowing into the trough at the top of the electrode overflowed in a thin smooth film, thus allowing better spacing of electrodes and less mercury inventory for cell operation.

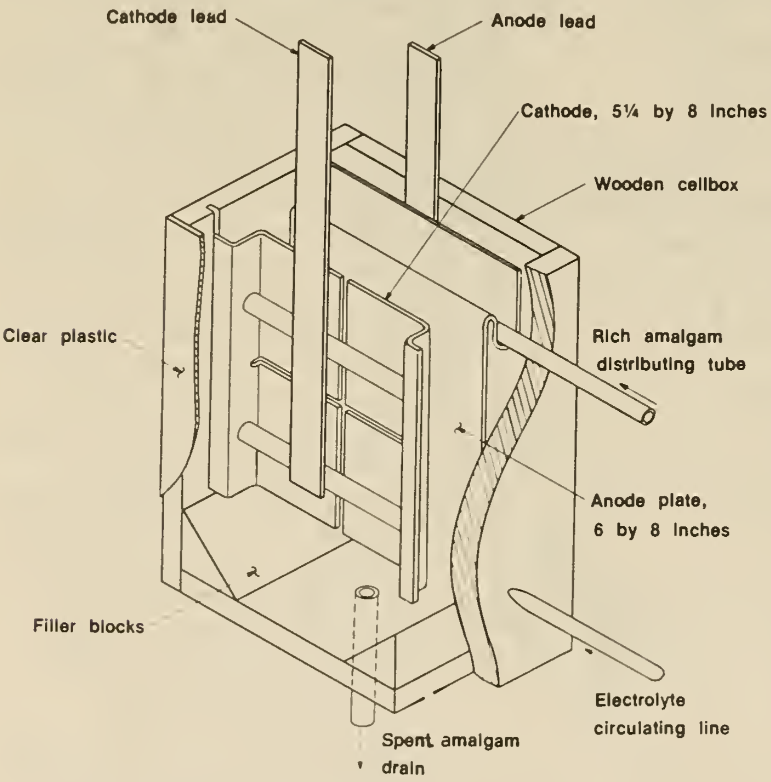


FIGURE 17. - Vertical amalgam electrode cell (87, 90).

The flowing amalgam was obtained by passing mercury over either galvanizers' dross or hardhead to selectively pick up zinc or tin prior to transferring the amalgam to the electrolytic cell. The cell provides an excellent means for electrolyzing high-purity zinc or tin from the amalgam film flowing down the anode over to the solid cathode.

Process Development

Scrap Iron Reduction of Nonmagnetic Taconite

A patented scrap iron-taconite reduction process, developed by Melcher (49) was found to be an effective and efficient method for reclaiming iron from a wide variety of low-grade iron scrap. The process offered dual advantages. First, a method for extracting iron values from abundant nonmagnetic taconite minerals that were normally set aside at a rate of 5 million tons annually as mining wastes; and second, the utilization of abundant low-grade ferrous materials such as junk automobile parts, tin cans, or mill turnings as reducing agents which were recovered as part of the reduction product. The basic chemical reactions comprised the reduction of the nonmagnetic iron oxide (Fe_2O_3) to the magnetic form (Fe_3O_4) in several reduction steps which may occur concurrently in various areas within a gas-fired kiln. The reactions (67) showing the utilization of scrap iron and fuel combustion products as reductants are as follows:

$$2Fe + CO_2 + H_2O \rightarrow 2FeO + CO + H_2,$$

$$6FeO + CO_2 + H_2O \rightarrow 2Fe_3O_4 + CO + H_2,$$

and

$$6Fe_2O_3 + CO + H_2 \rightarrow 4Fe_3O_4 + CO_2 + H_2O.$$

The metallic iron gains oxygen, and the hematite gives up oxygen with a net conversion of all iron constituents to magnetite. The overall reaction is

$$Fe + 4Fe_2O_3 \text{(taconite-hematite)} \rightarrow 3Fe_3O_4 \text{(magnetite)}.$$

Reduction efficiency was high, with nearly 95 percent of the taconite iron and virtually all of the scrap iron recovered in the product. The magnetic iron oxide concentrate contained 63 to 66 percent iron and 7 to 10 percent SiO_2. High recoveries and high-quality iron oxide concentrates were produced with other reductants such as household appliance scrap, municipal refuse, and sewage sludge. Since none of the products of reduction roasting are contaminated with processing chemicals and the tailings normally contain no phytotoxic materials, it would be possible to grow a cover of vegetation over the mill wastes generated by this process, thus helping to restore environmental quality at the mill site.

Wastes as Mutual Refining Agents

There are many metal-containing residues generated by the manufacturing sector of the metals supply and utilization system that are recovered by the secondary materials industry. Typical of these materials are the spent fluxes and residues generated in the utilization of zinc, specifically, spent protective flux cover mixtures for galvanizing vats and zinc-iron compounds which settle to the bottom of the vats.

In one typical investigation, Montagna and Ruppert (54) found that the spent flux material removed from the galvanizers' molten zinc bath could be used very effectively to remove 3 to 4 percent of the unwanted aluminum from molten zinc-base die-cast scrap. The zinc chloride in the "sal skimmings" ($ZnCl_2 \cdot NH_4Cl$ and ZnO) was reacted with the aluminum contained in the diecast according to the reaction

$$2Al + 3ZnCl_2 \rightarrow 3Zn + 2AlCl_3 .$$

The aluminum content of the resulting refined die-cast metal was reduced to 0.01 percent. The zinc metal released from zinc chloride during the reaction combined with the zinc melt, and the aluminum chloride was removed by simple volatilization.

Ruppert and Sullivan (72) developed a similar technique to create a solid phase of impurities in a molten metal solution, which was followed by simple filtration to separate the two phases. In particular, two metallic scrap materials were processed together as mutual refining agents to remove aluminum and iron impurities from the melt constituents. Since zinc-base die-cast scrap usually contains unwanted aluminum, and galvanizers' dross is comprised of zinc-iron intermetallic compounds with substantial amounts of free zinc, the two were melted together to form a solid aluminum-iron intermetallic compound in the zinc melt. The solid aluminum-iron compound was effectively removed from the melt by either filtration or centrifugation. Up to 92 percent of the total zinc in the original materials was recovered in a product containing only 0.02 percent iron and 0.20 percent aluminum. The residual aluminum was then removed to less than 0.01 percent by adding zinc chloride to the melt.

Physical Separation of Mixed Solids in a Rotating Spiral

Many problems are encountered in the preparation of scrap materials before processing. One typical example is the removal of insulating materials from aluminum or copper electrical conductors. For many years, the secondary plants had either burned or hammered the insulation off prior to melting. Often it was simply burned off during processing, such as smelting in the copper-scrap blast furnace. Sullivan (86) described a rather simple but effective spiral separator which, in effect, separated mixed constituents from scrap such as insulated cable that had been chopped into short sections and hammered to break away the insulation. A tilted rotating cylindrical device (fig. 18) was fitted with spiral ribs on the inside surface. The differences in the sliding friction of metal and nonmetal particles of the insulation-wire mix, for example, caused the metallic pieces to move in one direction parallel to the major axis of the device while the pieces of insulation moved in the opposite direction.

In a typical test, 9,743 grams of chopped and crushed rubber-insulated wire was passed through the separator. The recovered rubber fraction of 1,575 grams contained only 80 grams of metal and the 8,168 grams of metal product contained only 11 grams of rubber.

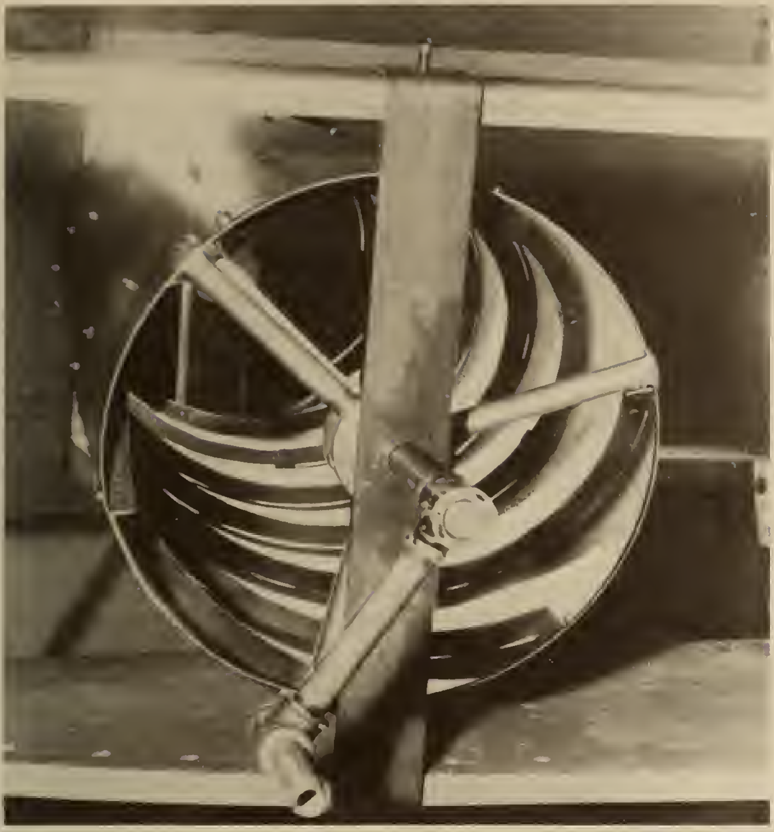


FIGURE 18. - Rotating spiral separator (86).

Analytical Tools for Scrap Sorters

Any process-development research involving the conversion of one type of material to another by removing or adding constituents, or by simply separating mixed constituents from each other, often requires rapid, convenient, and accurate analytical methods. The success of this type of research is critically dependent on the quality of the analytical data obtained.

A considerable part of the Bureau of Mines resource-recovery research pertains to the development of improved analytical techniques, procedures, and evaluations. One example of this effort involved the rapid identification of a wide variety of copper-base alloys for the purpose of developing faster and more accurate alloy identifications essential for scrap-sorting purposes. Campbell and Marr (16) conducted such an investigation in their evaluation of commercially available fluorescent X-ray equipment that could be used to analyze the common scrap alloys encountered by secondary brass and bronze processors. Their investigations included the positive identification of scrap copper-base alloys and accurate quantitative analyses. Three types of X-ray instruments were evaluated and compared for potential use by the secondary brass and bronze dealers, ingot makers, or foundries. The instruments included a conventional X-ray spectrograph, a portable X-ray spectrograph, and a radioisotopic X-ray analyzer using balanced filters. They were compared for rapid identification of scrap alloys and quantitive analyses needed to guide melting, blending, and costing operations in the secondary brass and bronze plants. Results indicated that both the portable X-ray spectrograph and radioisotopic X-ray analyzer held promise for identifying and analyzing the alloys. Although the alloys varied over a wide range of compositions, it was not necessary to make matrix corrections for any of the contained metals except copper. The conventional X-ray spectrograph was effective in the positive identification of all alloys examined and in quantitive determinations of the six elements of interest--aluminum, copper, iron, lead, tin, and zinc. Similar results were obtained with the portable X-ray spectrograph for those elements that produced wavelengths within the range limits of the instruments. This instrument distinguished between certain groups of alloys and provided quantitative determinations for copper, lead, tin, and zinc. The instrument sensitivity was marginal for analyzing small concentrations of elements if their atomic number was lower than 26. The investigation concluded that semiconductor detectors may also hold promise for sorting and analyzing copper-base alloys. Such applications may increase the use of dedicated computers to process analytical data and make processing decisions.

Maynard and Wilson (48) investigated the possibility of developing simple spot tests for identifying the major alloying elements in aluminum-base alloys. The tests were intended primarily for use by nontechnical personnel working outside the laboratory. Specifically, chemical spot-test procedures were to be developed for identifying zinc, copper, manganese, and magnesium, and chemical tests for identifying magnesium-silicon alloys and alumimum- and magnesium-base alloys. Simple devices and procedures were developed for identifying a wide variety of alloys by the color produced when a small spot of the alloy was reacted either chemically or electrochemically. The simple apparatus ultimately developed (fig. 19) was used for electrographic sampling (transferring a small spot of sample metal from the alloy to the treated paper). The anode clip was fastened to the alloy under test. In one version of the identification procedure, the treated sample paper was placed over a suitable area on the alloy specimen, and the aluminum cathode was pressed against the other side of the paper, which had been wetted with a few drops

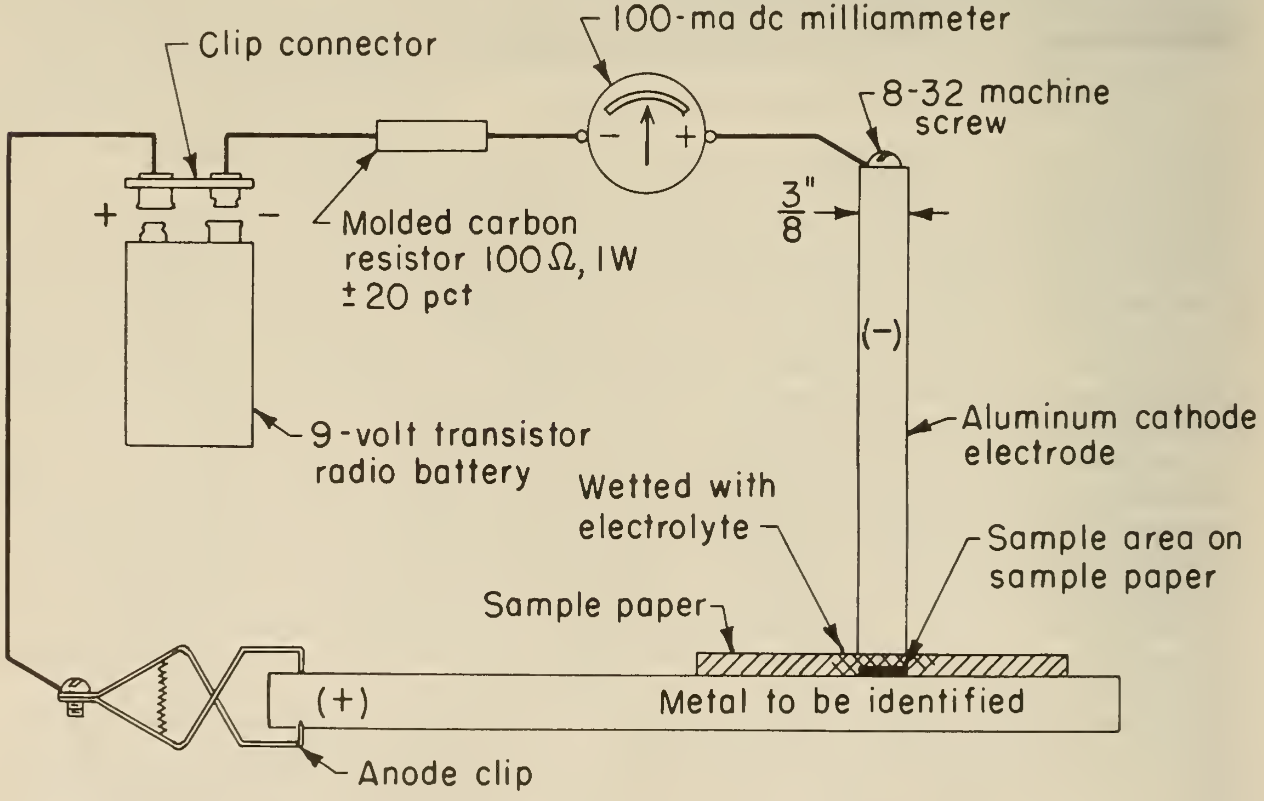


FIGURE 19. - Spot-testing equipment (48).

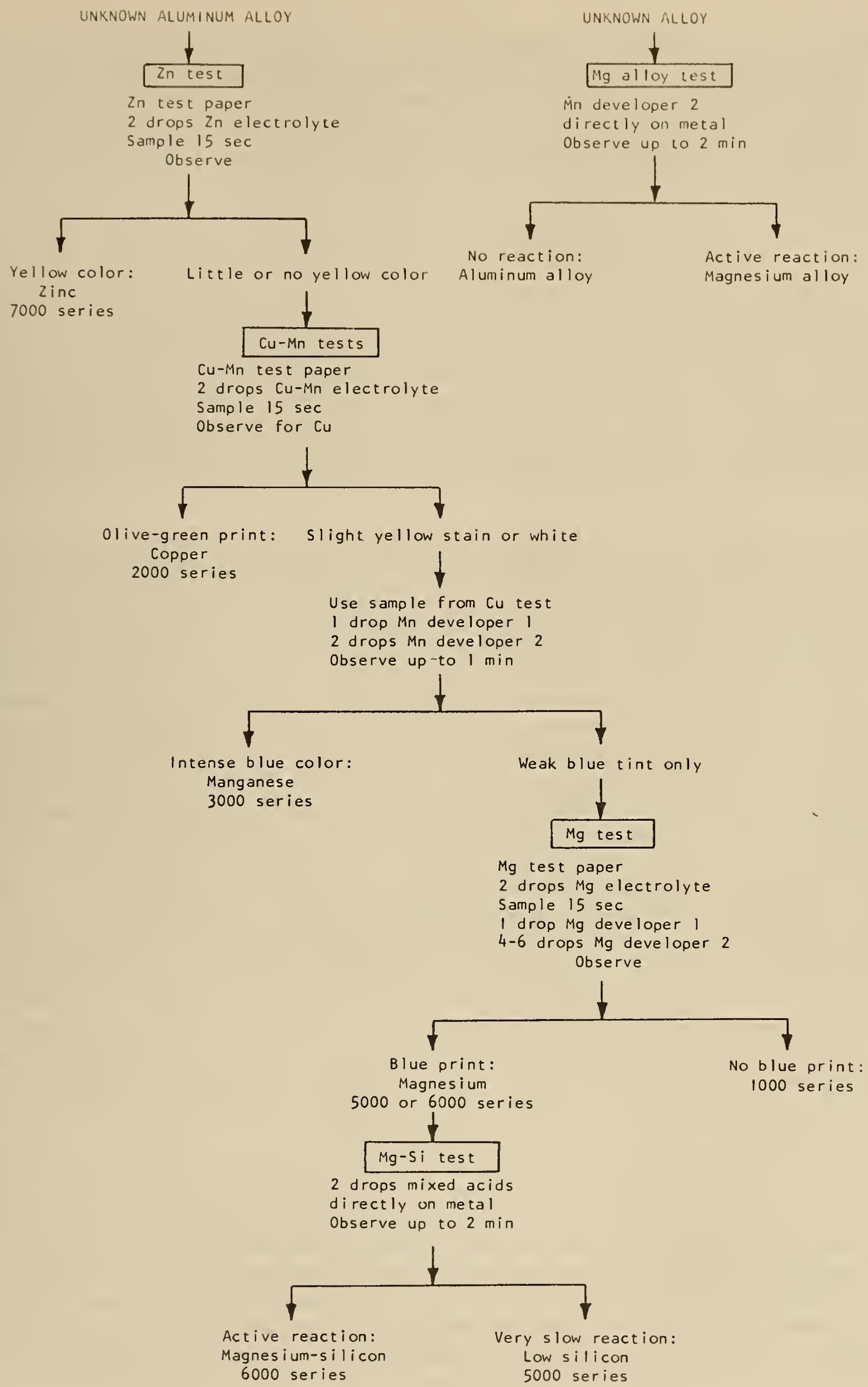


FIGURE 20. - Chart for spot-testing procedures (pretreated papers) (48).

of suitable electrolyte. The alloy sample was transferred to the paper collector during a few seconds of electrolysis. The paper was then removed, and a developer solution was applied, when necessary, to produce an identifying color. Figure 20 illustrates the procedures for making a series of identifications with treated papers. Similar procedures were developed for identification with untreated-paper sample collectors.

POSTCONSUMER MATERIALS--UTILIZATION AND DISPOSAL

Adjusting 1971 data (44) on the basis of a 3-percent increase per year indicates that more than 210 million people in the United States are collectively generating about 145 million tons of urban refuse annually. This amounts to a total of nearly 800 million pounds per day, or nearly 4 pounds per person per day. Based on conversations with many municipal officials, the estimated average cost for the collection and disposal of municipal refuse is about \$20 per ton. Officials from some of the larger cities reported costs of \$40 per ton for this service as early as 1970.

In recent years, the problems attending urban refuse disposal were largely responsible for a multitude of new attempts to solve the disposal problems by developing new technology for recovering values from these wastes and developing more efficient, less expensive methods for the disposal of nonrecoverable fractions. The number of publications on these activities has increased markedly during the past 5 years, appearing prominently in nearly all of the literature on resource recovery (3-4, 77-79). Many of these publications and reports pertain to characterizations and analyses of the refuse and the collection and disposal systems to improve recovery utilization and disposal processes (81-82). Others have investigated the economics of prototype processes for extracting values from municipal refuse based exclusively on conceptual arrangements of mostly conventional equipment to yield products of assumed grade and value. A comparison was made (1) of the economic benefits in systems that would only recover noncombustible materials (metals and glass) with those benefits derived from systems that would recover noncombustibles and energy from the combustibles. It was concluded that favorable economics of such systems must be based on more than one source of revenue. It was also concluded that the addition of energy recovery to the basic process would benefit most communities. Other evaluations revealed many uncertain factors that could alter the overall economics of any given system unpredictably. This supports the common concept that waste disposal problems are significantly different in each municipality and each one should consider its own situation as unique.

There are only six general types of refuse disposal and utilization practiced in the United States: ocean dumping, open dumping on land, open dump burning, landfilling, incineration, and resource-recovery-disposal systems. The first three are presently banned in the majority of municipalities; total ban in the United States should be in force in the relatively near future. Landfilling is the most extensively practiced disposal method, but large amounts of refuse are still incinerated. Fortunately, energy is recovered at some incinerators.

The Bureau of Mines has been active in recovering resources from refuse since the Solid Waste Disposal Act of 1965 was passed. Ultimately, two pilot plants were built and operated successfully. Both are still operating intermittently. The first was designed to recover the valuable constituents from incinerator residues (36) and the second for recovering values from raw municipal refuse (90). The flow diagrams for the incinerator residue and raw refuse plants are shown in figures 21 and 22, respectively. Sullivan and Makar have recently prepared a comprehensive report (93) on the quality of products (tables 7-8) recovered in these systems and their suitability for recycling. Products were examined extensively by the Bureau of Mines and in other laboratories and industrial firms. In nearly all cases, the materials were found to be perfectably suitable for many new uses. The quality of some of the products compared favorably with similar secondary raw materials for which there has been a heavy demand historically; in many instances, the products derived from refuse were of superior quality. The authors concluded that the emerging resource recovery industry should be encouraged by the promising climate in the marketplaces for recovered products that will be acceptable for recycling.

TABLE 7. - Pilot-plant products obtained from typical incinerator residues (89)

Product	Percent
Large ferrous metal	15.0
Fine ferrous metal	5.0
Aluminum	1.75
Heavy nonferrous metal	.75
Clean glass	30.0
Slag	25.0
Fine ash and tailings from mineral jig and flotation cells	22.5
Total	100.0

TABLE 8. - Typical pilot plant products obtained from raw refuse (89)

Product	Percent
Light combustibles (fuel)	59.3
Ferrous metal	7.6
Aluminum	.8
Heavy nonferrous metal	.2
Glass	10.5
Heavy combustibles (fuel)	5.0
Putrescibles dewatered to 50 percent solids (fuel)	6.6
Fine glass, grit, dirt, ceramics	10.0
Total	100.0

Although there had been few attempts to recover marketable products from refuse prior to 1965, the scope and diversity of recycling methods now under consideration are markedly greater. It now appears that the number of basically different processing concepts has reached a maximum, but the level of development on several types of systems continues to increase. This trend will probably continue during the next decade.

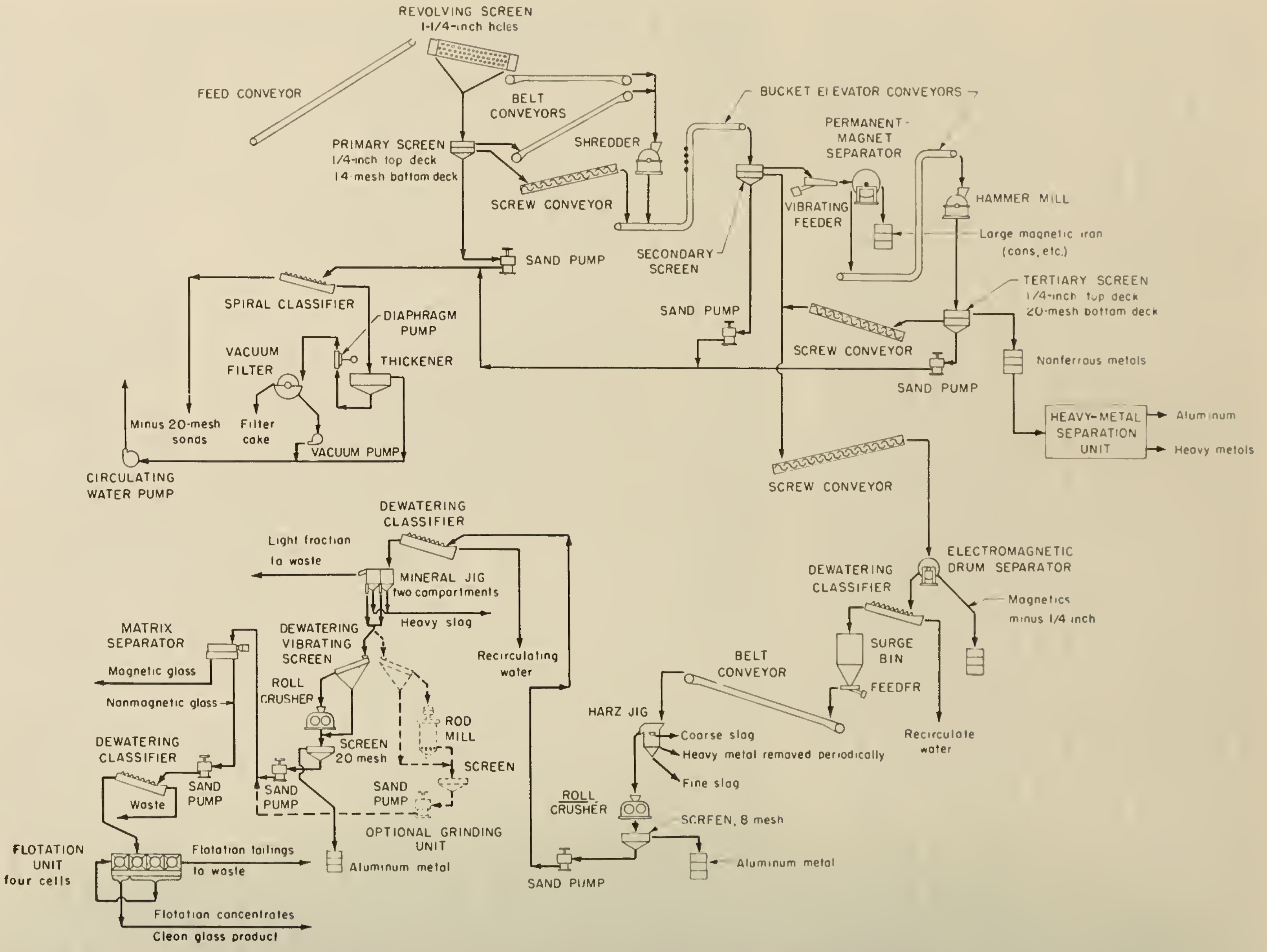


FIGURE 21. - Incinerator residue recovery flowsheet (89-90).

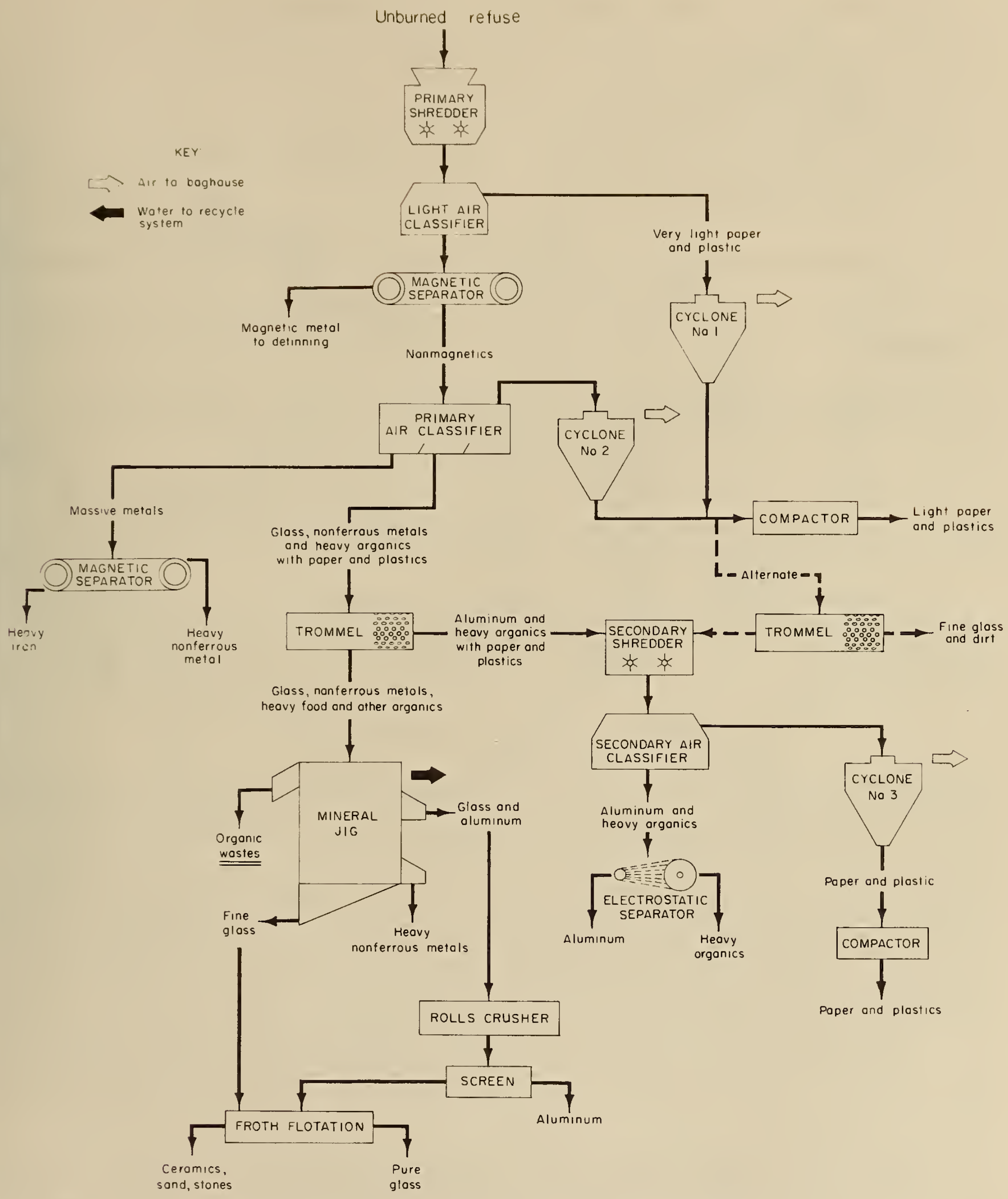


FIGURE 22. - Raw-refuse recovery flowsheet (89-90).

The three basic refuse incineration systems include--

1. Incineration and disposal of residues.

2. Incineration with energy recovery only.

3. Incineration with energy and resource recovery.

The four basic raw refuse systems include--

1. Recovery of combustibles for direct use as fuel.

2. Recovery of combustibles and conversion to fuel products (solid fuel, oil, methane).

3. Recovery of combustibles and separation into fuel and nonfuel products (paper for fiber or fuel, putrescibles for fuel or direct conversion to animal food, plastics, and other combustibles for fuel).

4. Optimum recovery of all values (paper, plastics, and organics for fuel or recycling; ferrous metals, copper-zinc metals, aluminum-base metals, flint glass, amber and green glass for recycling).

The Bureau of Mines research program has included projects relating directly or indirectly to all of the preceding systems for processing raw or incinerated refuse. The following discussions pertain to selected typical investigations.

Plastics From Processed Raw Refuse

Holman, Stephenson, and Adams investigated a variety of methods for separating the mixed plastic constituents of urban refuse into products of suitable quality for recycling (37). Materials for the investigation were obtained from a local collection drive, a refuse collection center, and the Bureau of Mines refuse-processing pilot plant. Separation methods were also applied to the upgrading of chopped-wire insulation wastes produced at secondary-metals plants where chopped aluminum and copper wire scrap is processed. A number of related studies were made of potential methods for obtaining marketable products from vinyl plastics, and procedures for fabricating waste thermoplastics into useful products. For the purpose at hand, discussions are confined to the type of experimental separator illustrated in figure 23. The system included a sink-float separation followed by elutriation separation. An air bubble lift column raised the final plastic fraction to a collection screen.

A mixture of chopped plastics consisting of polyolefins, polystyrene, and polyvinyl chloride was fed into the water sink-float section where the heavier materials sank to the bottom of the column, but the polyolefins were floated out of the top of the tank onto a screen-bottom collecting bin. The sink portion, which was polystyrene and polyvinyl chloride, was airlifted to the top of the elutriation column. The polyvinyl chloride sank to the bottom of

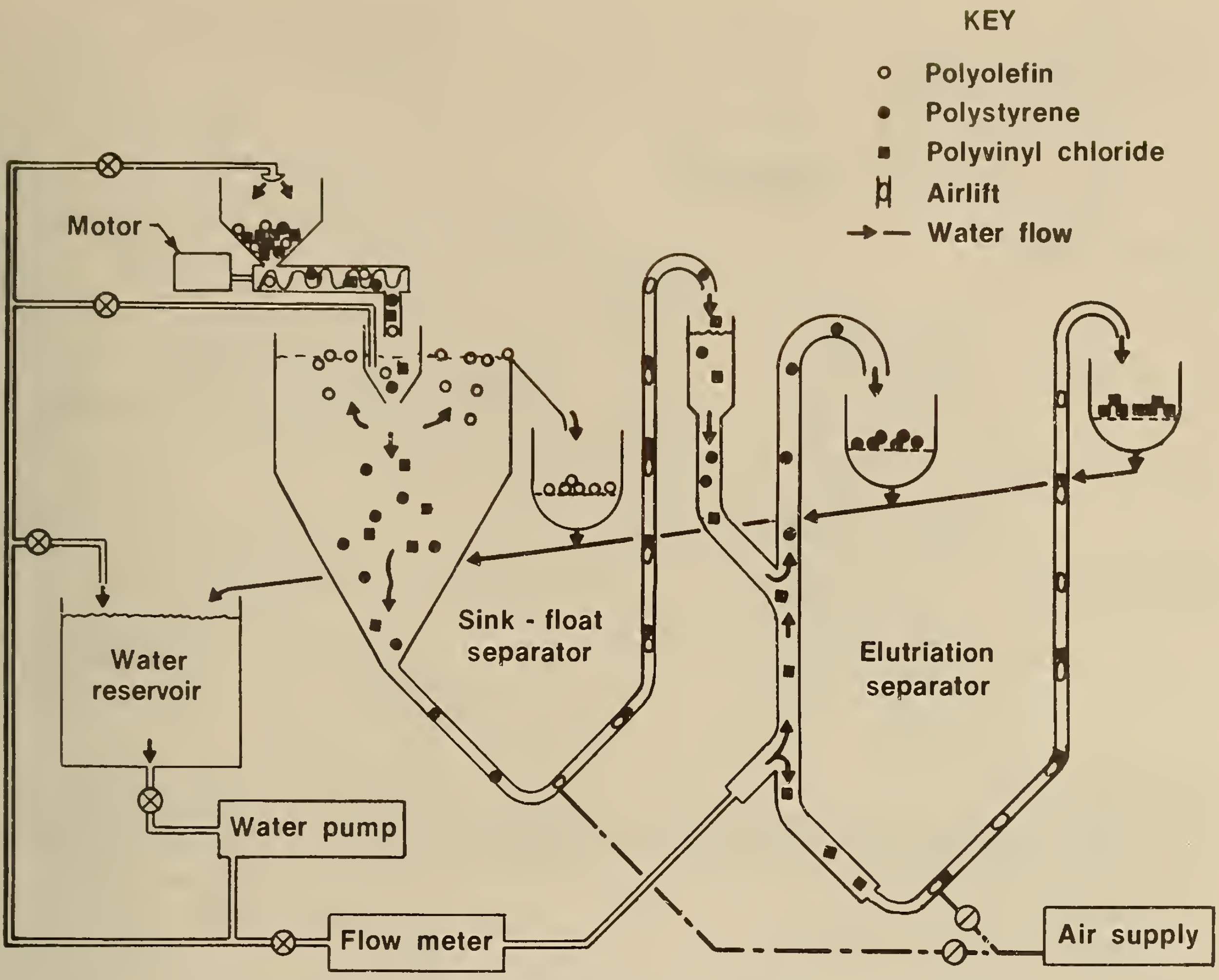
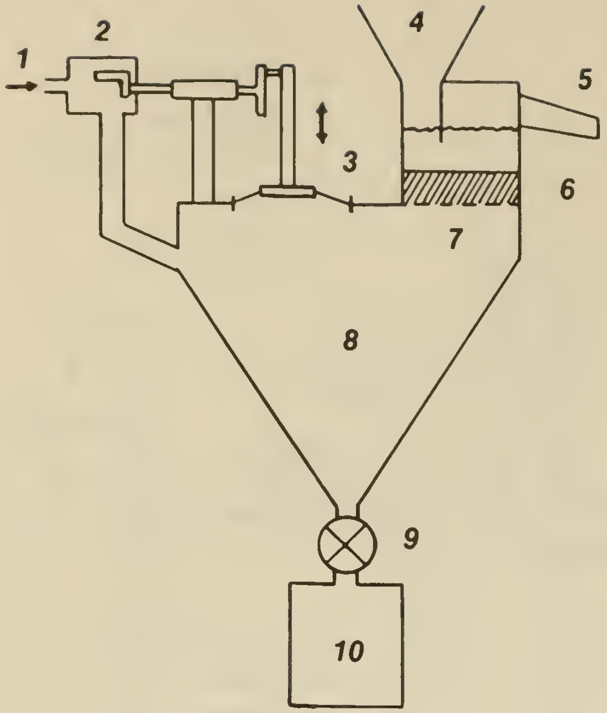


FIGURE 23. - Hydraulic separation of mixed plastic particles (37).

the column through a rising flow of water in the elutriation column, which carried the polystyrene over the top of this column and into a screen-bottom collecting bin. The polyvinyl chloride particles were then airlifted into the last collection bin. It was found that separation of plastics by hydraulic methods is enhanced considerably by the addition of 0.3 cubic foot of surfactant per gallon of water. The rates of separation and the level of product purity are sufficient to justify the design of a prototype commercial-scale unit for urban-refuse-processing plants.

The well-known mineral jig (fig. 24) was also found to be quite effective in an unconventional separation of mixed metal and plastic components of chopped electrical conductor and plastic insulation particles (37). The operation is based on the behavior of a rapidly pulsing liquid-solid bed. The mixed particles are fed into the pulsating system above a bed of gravel, which rests on a screen at the bottom of the bed. The pulsing action introduced near the surface of the system repeatedly forces the gravel bed apart, thus allowing the heavier particles of metal to sink through the bed of gravel and



1 Water inlet
2 Rotating water valve synchronized with pulsator
3 Rubber diaphragm pulsator
4 Material feed
5 Water and tailings overflow
6 Bed
7 Grizzly
8 Hutch (water container)
9 Hutch valve
10 Metal concentrate

FIGURE 24. - Mineral jig (37).

the supporting screen. The heavy fraction then collects on the bottom of the tank. Water, fed continuously into the unit, carries the light fraction (plastic) out of the system overflow. It was found that copper could be removed from wire insulation wastes in concentrations up to 98.5 percent. Almost all copper concentrates exceeded 80 percent. In one case, aluminum was recovered from waste insulation in a concentrate of 99.4 percent.

Electronic-Optical Sorting of Colored Glass

While conducting research on the recovery of valuable constituents from raw municipal refuse and from the residues from incinerated municipal refuse (89-90), the Bureau of Mines found that glass accounts for 8.5 percent of the former and about 45 weight-percent of the latter. The glass fraction recovered from these materials during processing is comprised of fragments of many different colors with some ceramic and rock particles present. Mixed colored glass contaminated with small amounts of ceramic impurities is generally less valuable for recycling than if the glass is first separated into lots of individual color and free of all nonglass impurities and has a narrower market. The commercial electronic color-sorting machine illustrated in figure 25 was evaluated by Palumbo, Stanczyk, and Sullivan (61) for its potential application in the Bureau of Mines systems for processing the glass fraction of urban refuse (90).

In operation, pieces of feed material are discharged from a hopper onto each of two belts that carry the pieces in single file and propel them into a fall pattern that passes the streams of glass pieces, in very rapid succession, through a pair of optical examination units. Each examination section is

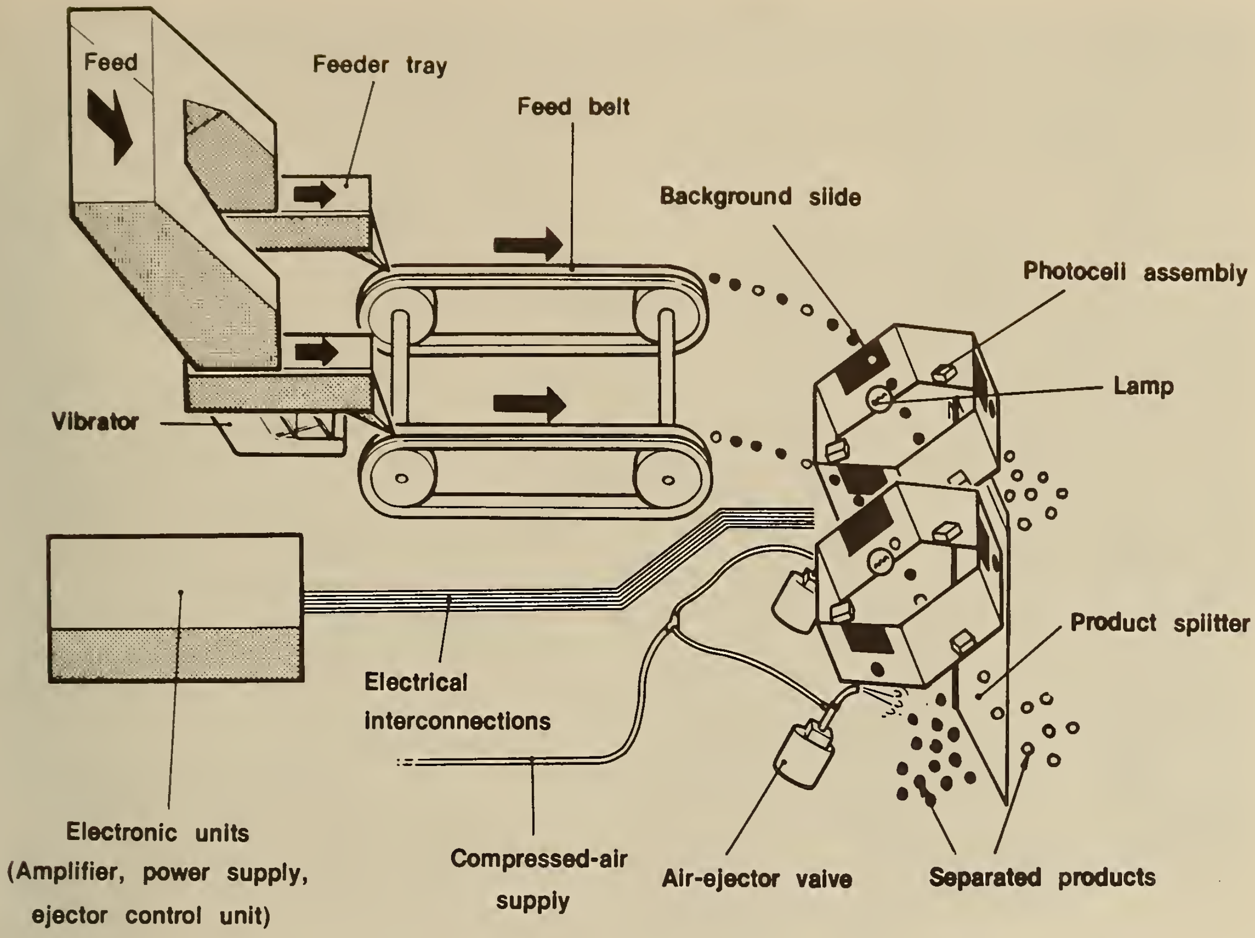


FIGURE 25. - Optical color sorter. *(Courtesy, Sortex Co., N.A., Lowell, Mich.)*

fitted with three high-intensity lamps to illuminate the falling particles. Three photoelectric cells are set 120° apart with optics arranged to give a full 360° view of the individual particles falling by. Different light filters can be inserted over the photocell lenses to enhance the reflective qualities of the particle. Standard background color plates are located directly opposite each photocell to create a null response from which any change is detected and signaled to the electronic section, where desired operational parameters can be set in (such as color contrasts and time delay of a few milliseconds) to allow the particles time to fall into the ejection zone. If the settings are adjusted for ejection of dark-colored particles, for example, the ejection valve will be activated when each dark particle is within range of a high-pressure air jet actuated by the photocell sensors. The air jet striking the dark particles changes the direction of their fall so that they can be collected in a separate container apart from the other particles which fall freely through the photocell system.

With proper adjustment of the equipment feed rates and preparation of feed material, the following separations were achieved:

1. An amber glass fraction of 90 to 100 percent amber and 0 to 10 percent of flint and green glass combined.

2. A flint fraction of 95 to 100 percent flint with 5 percent or less of amber and 1 percent or less of green.

3. A green fraction of 50 to 100 percent green, 15 percent or less of flint and 35 percent or less of amber.

The foregoing meets color specifications for purchased cullet in the United States, but no attempt was made to develop economic data for this specific operation.

Electrodynamic Separation of Refuse Constituents

Substantial quantities of paper and plastics are recovered during the processing of urban refuse, but this fraction is usually contaminated by other materials such as glass, dirt, organics, and by the mixing of plastic and paper constituents. An important study was made by Grubbs and Ivey (33) to help develop a method for processing such materials into a paper-free plastics fraction and a plastics-free paper fraction. They found that good separations could be made using equipment illustrated in figure 26. In operations for separating paper and plastic, the mixed fragments were fed onto a large metal drum that was rotated at an appropriate speed to carry the fragments into the corona field that was formed between a wire electrode at a distance and parallel with the axis of the drum. The drum was operated at a negative potential of up to 45 kilovolts. As the fragments of paper and plastics moved into the electrostatic field, plastic fragments clung to the drum, but paper fragments were drawn toward the wire electrode. The plastic was brushed off the bottom of the drum. A splitter was adjusted to separate the recovered paper and plastic fractions into their respective collection bins. The paper and plastic fragments behaved differently because of their different dielectric properties. Accordingly, as would be expected, moisture influenced the properties of paper much more than plastics. It was found, for example, in operations at 10 percent moisture, that plastic concentrates contained 68 percent plastic and 32 percent paper. The paper concentrates was 100 percent paper. The plastics concentrate improved with increasing moisture up to 55 percent, at which point both the plastic and paper concentrates were 100 percent. No plastics appeared in the paper concentrate within the moisture range of 0 to 55 percent. In operations with refuse materials, mixed plastics concentrates analyzing 99.4 percent plastics were recovered consistently from the light material fraction of shredded refuse. Corresponding paper fractions analyzed 99.9 percent paper. Recovery of plastics was consistently better than 99 percent. It is doubtful if laboratory results could be achieved on a commercial scale; however, if the major part of the paper could first be recovered as fiber by pulping, then the plastic-rich residue might be economically separated.

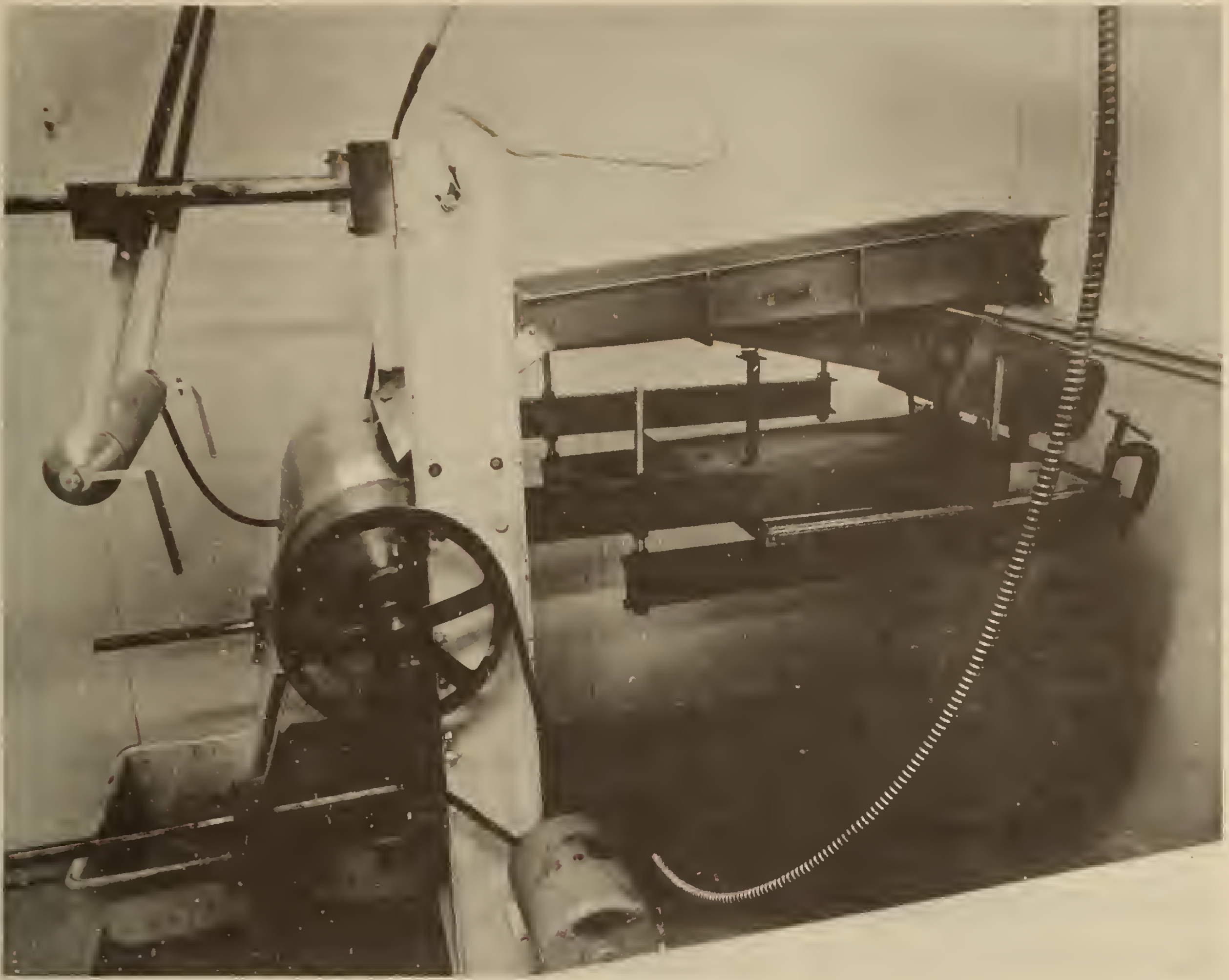


FIGURE 26. - Electrodynamic separator (33).

Recovery of Waste Lubricating Oil

Each year, the U.S. service stations, airfields, railroads, trucking, and bus maintenance and repair shops dispose of more than 1 billion gallons of spent lubricating oil, most of which might be profitably reclaimed by new rerefining techniques. Whisman, Goetzinger, and Cotton (101-102) developed and tested several techniques for reclaiming and testing used crankcase oil, some of which were promising. They included (1) treatment by percolating through both ion-exchange columns and beds of chemically treated clay, (2) distillation through wiped-wall vacuum apparatus, (3) solvent-extraction systems, and (4) removal of impurities by chemical and chelation treatment. The objective was to evaluate various systems for refining used crankcase oil to a quality comparable to that of virgin oil.

Percolation through caustic treated clay, the simplest technique tried, would not effectively reduce the level of contained inorganics, additives, or metallic contamination. Pretreatment with an alcoholic solvent followed by treatment with a strongly basic anion-exchange resins proved to be most effective in removing acidic components, but was less effective in removing the metallic materials. A strongly acidic cation exchanger would not effectively remove the acidic components left in the residual following alcohol treatment. This suggests that the treatment in two ion-exchange columns, each packed with monobed sections, may provide efficient polishing of solvent-treated lubricating oil.

Distillation in a continuous vacuum still produced a rerefined lubricating oil closely approaching the quality of the original base oil, but the processing was complicated by the viscous residue that was generated during distillation. Best results were obtained using a combination of solvent treatment with alcohol prior to vacuum distillation into a continuous wiped-wall still. Hydrocarbon composition stability of the product was high, and recompounding with a complete additive package produced rerefined oil of a quality that passed all laboratory tests. Results of experiments using solvent treatment followed by reaction with a chelating agent indicated very successful removal of metallic compounds from the waste oil, but the cost of this technique appeared to be too great for further practical consideration.

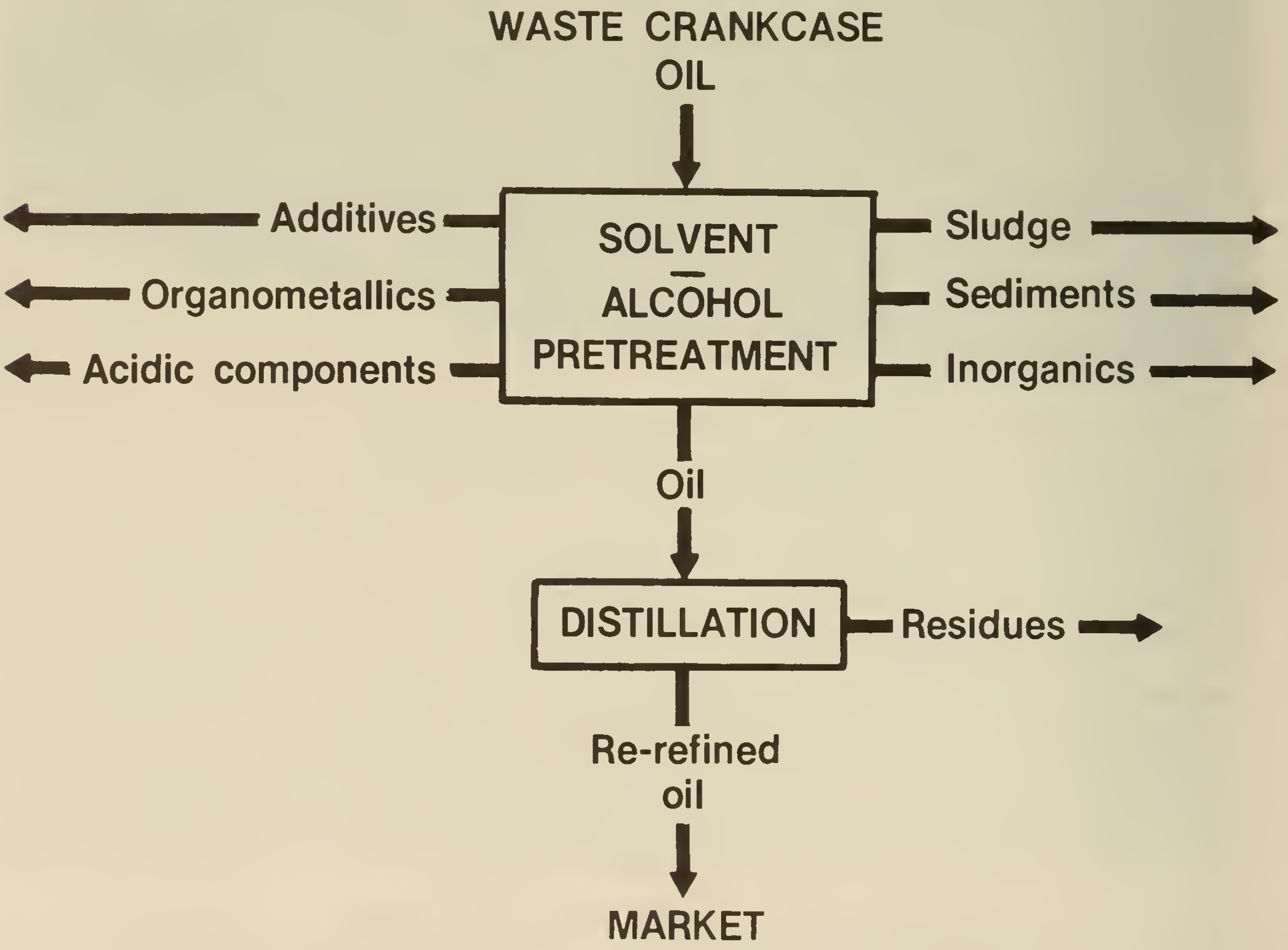


FIGURE 27. - Recovery of waste lubricating oil (101-102).

The process having greatest potential for commercial application is illustrated in simple form in figure 27.

Pyrolysis of Scrap Tires

By 1968, the rate of used tire accumulations in the United States had exceeded 200 million per year. Replacement of these tires consumed 5.5 billion pounds of new natural and synthetic rubber and more than 400 million pounds of fabrics (71). Since relatively few used tires are retreaded, tremendous numbers must be disposed of, causing ever greater environmental problems and complicating the waste disposal situation.

In a concerted effort to solve disposal problems, the Bureau of Mines, in cooperation with the Firestone Tire and Rubber Co., Akron, Ohio, initiated pyrolysis studies on the feasibility of carbonization (destructive distillation) by methods that would solve the pollution problems and yield marketable products (104).

Tire scrap was charged into an electrically heated pyrolysis unit illustrated by the simplified version of the process in figure 28. The retort could be heated to temperatures up to 900° C depending on the nature of the test. Retort effluents were subjected to a train of consecutive treatment sections designed to collect the volatilized constituents as separate products. It was found that the amount of the various products was directly related to the temperature of carbonization. The following examples are typical of the test results. For each ton of charge processed at 500° C, the yield was 800 to 896 pounds of residue, up to 128 gallons of oil, and 1,720 cubic feet of gas. At 900° C, the yield was 1,172 pounds of residue, 51 gallons of oil, and 12,630 cubic feet of gas. Nearly all of the sulfur remained in the residues regardless of temperature. After evaluating the operational parameters and the quality and yield of products, it was concluded that the process is technically feasible as a commercial process and all products are potentially valuable.

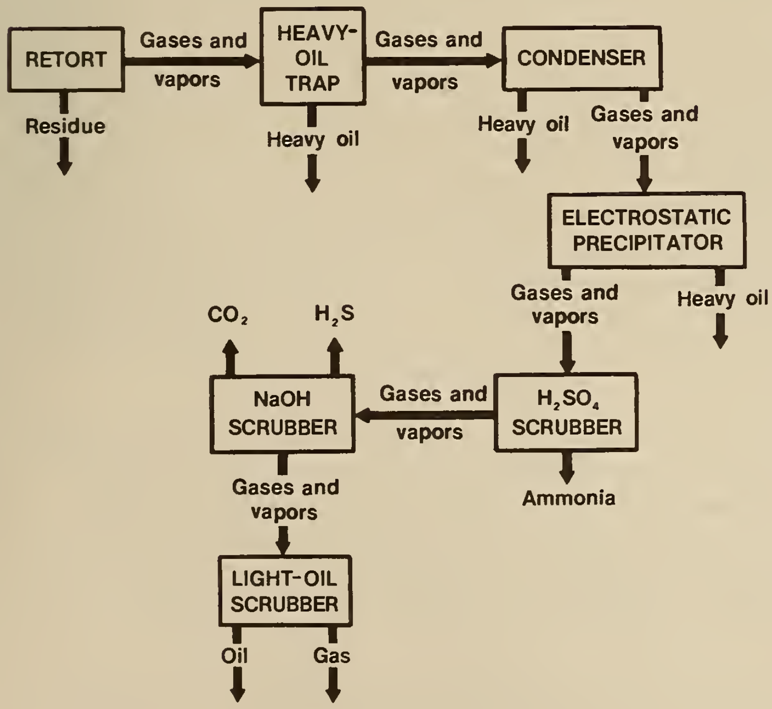


FIGURE 28. - Pyrolysis of scrap auto tires (71).

The Bureau of Mines conducted similar tests on the conversion of animal wastes, industrial wastes, and urban refuse constituents to gaseous and liquid fuel with equally encouraging results (85).

CONCLUSIONS

Since the scope of this paper does not permit a complete description of all research in the field, additional information is available in reports on other resource recovery research projects listed under supplementary references.[3]

Accomplishments in this area of research dictate a strong need to continue and expand the effort. The goods and materials manufactured today will be available for recycling during the coming years, and the physical character and composition of most manufactured devices and materials will change constantly with the continued growth of the economy, an increase in the standard of living, and industrial progress. Additional changes in types and quantities of waste will accompany industrial compliance with the Environmental Protection Agency standards that must be met by July 1977 and even with the more rigorous standards that must be met by July 1983. More bulky wastes will be generated and much greater quantities of materials such as utility plant fly ash, mill tailings, and plastics manufacturing wastes will be produced. Large quantities of sludges from chemical processes and sulfur dioxide scrubbers will be collected for processing or disposal; however, waste disposal costs will mount as disposal areas become scarce. Wastes produced by new processing and manufacturing industries will add to the already difficult disposal and processing problems. Since this situation will probably prevail for many decades, resource recovery research is far from complete.

Many problems in this field of research are not entirely technical in nature. Among the major problems in planning future research programs is the great need for information on the composition, quantities, and locations of many industrial wastes presently being disposed of in virtual secrecy. This "quiet disposal" is due primarily to widespread fears of bad publicity and costly restraints if such disposal becomes common knowledge. There is little doubt, however, that a complete accounting of all such wastes would be of tremendous value in planning future research. Finding and identifying such wastes in terms of respective compositions, quantities produced, and locations is perhaps one of the most important future tasks. The establishment of the St. Louis Waste Exchange is an important first step.

Another important problem is the long span of time (5 to 20 years) that normally passes between the event of new discoveries and the date of their commercial application. This lapse of time is usually attributed to the reluctance by industry to suffer the loss of earlier investment costs for facilities and equipment that would no longer be useful in the new processes and the high cost of new equipment, processes, and facilities. Greater incentives are needed to promote a faster changeover to improved industrial methods and devices after they become available. One incentive would be to develop more effective and more conclusive ways to demonstrate the technological and economic advantages of all newly developed processing alternatives as they become evident.

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[3] To obtain copies of Bureau of Mines publications, send requests to Publication Distribution Branch, Bureau of Mines, U.S. Department of the Interior, 4800 Forbes Avenue, Pittsburgh, Pa. 15213.

The examples of research in the foregoing pages and other research cited in the selected bibliography demonstrate the fact that new innovative research offers excellent opportunities for significant advances in the technology of recycling. But only through extensive communications can the knowledge gained be put to optimum use. This emphasizes the importance of frequent and meaningful symposia and publications as research programs continue. Achievements to date indicate a high potential for the emergence of a thriving waste utilization industry during the next decade.

The collective wastes presently being discarded comprise a vast rejected resource. No industrialized nation can continue to reject such a resource indefinitely. Its ultimate use is therefore inevitable.

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